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ORIGINAL RESEARCH

Measuring dimensional stability of thermally modified ayous and iroko wood: comparison of the three test methods

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Abstract The dimensional stability and moisture content of wood are important physical properties that influence the end-use of wood. These properties can be greatly improved by thermal modification of wood. In this study, three different test methods are studied on thermally modified iroko (*Milicia excelsa*) and ayous (*Triplochiton scleroxylon)* wood to compare the methods of measuring dimensional stability behavior. Test methods include (1) repeated water-soaking until saturation, (2) test cycles of water-soaking and drying, and (3) exposure to relative humidity cycles in the air. Test methods revealed enhanced dimensional stability in thermally modified wood samples relative to the unmodified samples. However, the relative dimensional stability behavior varied between the methods. Water-soaking until maximum swelling (Method-1) showed differences in the swelling rate for each wood material. Thermally modified ayous wood swelled more slowly (~44%) than thermally modified iroko wood. Interestingly, subsequent test methods (Methods 2 and 3) showed decreasing dimensional stability especially for modified iroko wood as the wetting–drying cycles increased and iroko wood was kept in high relative humidity such as 85%. The cracks in the cross-section were more in modified iroko samples than in the controls, whilst modified ayous wood had no visible cracks in the cycles of wetting and drying in Method-2. The anti-swelling efficiency of thermally modified ayous wood was found as ~45%, 30%, and 40–60% in Method 1, 2 and 3, respectively. The results of the three different methods clearly showed that thermal modification ensured good dimensional stability for ayous wood, whereas in iroko wood insufficient dimensional stability was obtained, especially in Methods 2 and 3. Thermal modification may not be a good option to ensure dimensional stability for iroko wood. For a complete understanding of dimensional stability in wood modification studies, long-term wetting and drying cycles and/or air humidity cycles are recommended especially for extractive-rich wood species.

Keywords Thermal modification · Dimensional stability · Air humidity · Water absorption · Wood · Swelling

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Introduction

Wood is a hygroscopic material due to several hydroxyl groups (OH^-) in the cell wall polymers, thus

wood can change its dimensions due to the absorption of water vapor and capillary water. These dimensional changes are anisotropic and are greatest in the tangential direction, then in the radial direction, and least in the longitudinal direction (Hill 2006). One of the key properties that influence wood's suitability for various applications (such as doors, windows, floors, etc.) is its dimensional stability, which relies on how much it shrinks and/or swells with changes in moisture content (Sargent 2019).

Thermal modification improves the dimensional stability and durability of wood and reduces hygroscopicity and wettability depending on the treatment conditions (Kocaefe et al. 2008; Juodeikiene 2013; Lekounougou and Kocaefe 2014; Li et al. 2017; Zhang et al. 2023). Improved dimensional stability in thermal modification can be achieved by the following items, (1) the presence of bulking agents within the cell wall, (2) the removal of OH^- groups, (3) increased crosslinking within the cell wall polymers, and (4) changes in the mobility of the polymer network (Hill et al. 2021). Anti-swelling efficiency (*ASE*) which shows the dimensional stability of wood increases both with temperature and treatment time (Hill 2006; Priadi et al. 2019; Gennari et al. 2021a; Cao et al. 2022). The equilibrium moisture content (*EMC*) of ThermoWood®-D class is approximately 40–50% lower depending upon the species modified and the exact conditions employed for the treatment at a given relative humidity (RH) compared to controls (Hill 2006). In Türkiye, thermally modified iroko, ayous, ash, spruce, pine, tulipwood and oak are industrially available wood species but among them, thermally modified iroko wood and thermally modified ayous wood have been extensively used as construction materials. Thermo iroko wood is mostly preferred as deck and exterior cladding, whilst thermo ayous wood is preferred in facade applications. The estimated annual production (m^3) of thermally modified wood is 535.000 m^3 in Europe (Jones et al. 2019) while it is approximately 40.000 m^3 in Türkiye. The production volume of thermally modified ayous and thermally modified iroko wood is estimated to be approximately 12.000–15.000 m^3 per year.

Assessing the dimensional stability of wood is crucial to comprehend the behavior of modified wood in a variety of settings and applications (Sargent 2022). Indeed, some research papers have compared the dimensional stability of thermally modified iroko and ayous wood samples, and found 13% *ASE* with 4.6% decrease in density, and 18% *ASE* with 1.3% mass loss for thermally modified iroko wood (Korkut 2012; Icel and Beram 2017), and 51–66% *ASE* for thermally modified ayous wood with a decrease 11–16% in density (Gennari et al. 2021a, b; Gennari et al. 2022). The results were particularly affected by the thermal modification conditions, dimensional stability test procedures, and variations between stands of trees of the same species. Comparative performance between different wood species may be dependent on the test methods used for measuring the dimensional stability of wood. However, as far as known, there is no standardization on test procedures for dimensional stability measurements of modified wood, and there is little consensus on appropriate test procedures (Sargent 2019, 2022). Sargent (2019) reviewed dimensional stability measurements in solid wood and classified the dimensional stability methods into two basic underlying principles as (1) contact with liquid water and, (2) contact with water vapor until equilibrium is reached, or a shorter duration. The membership of the Thematic Network for Wood Modification grouped different test methods as follows: (1) dimensional change due to water soaking/impregnation (2) dimensional change under a range of relative air humidity conditions (3) cyclic wetting and drying, and (4) altered *EMC* and the rate to reach this point (Van Acker 2003).

Among the dimensional stability tests involving liquid water, the cyclic water-soaking and drying test method that was suggested by Rowell and Ellis (1978) is the most popular test method for wood modification studies (Van Acker 2003; Hill 2006; Ermeydan et al. 2014, 2021; Cermak et al. 2016; Hill et al. 2021; Sargent 2022; Kupfernagel et al. 2023; Liu et al. 2024a) but not the only method consistently used. Dimensional stability measurements of modified wood based on the submerge in water until saturation (ISO 4469 1981; ISO 4859 1982; BS ISO 13061-14 2017, ISO 13061-15 2017, ISO 13061-16 2017; Schorr and Blanchet 2020; Gennari et al. 2021a, b, 2022; Olaniran and Militz 2024; Liu et al. 2024b) or water submersion tests in different periods (Temiz et al. 2006; GB/T 1934 1-2 2009a, b; Tomak et al. 2011; Dubey et al. 2011; Korkut 2012; Jiang et al. 2015; Dong et al. 2016; Icel and Beram 2017; Priadi et al. 2019; Chen et al. 2020; Demirel Kose 2023; Kaya 2023; Can et al. 2024; Dong et al.

2024; Zhao et al. 2024) are also common procedure for solid wood. Maximum swelling of the wood after water vacuum-pressure impregnation (vacuum: 20 hPa, 30 min; pressure: 0.8 MPa, 30 min) and subsequent water storage at 60 °C for 24 h was also examined the change in dimensional stability (Welzbacher et al. 2007). The dimensional stability of wood according to the GB/T 30364-2013 method, in which samples were boiled for 4 h, dried at 63 ± 3 °C for 20 h, and boiled for another period of 4 h was studied by Sun et al. (2024). There are many variations in these test methods such as sample sizes, especially the size of cross-sections, number of water-soaking and/or drying cycles, soaking time, and some possible drawbacks due to the drying ends with cracks on cross-sections. Beside these methods, the swellometer test (ASTM D4446 2013) and AWPA E4 (2021) for measuring short-term dimensional changes and water repellency (30 min) are also used for wood (Schmidt et al. 2002; Khalil et al. 2013; Demirel Kose et al. 2018). The swellometer test which measures the tangential swelling after 30 min has some limitations in practical use since a wood type that swells very little but quickly could look the same as a wood type that swells a lot but very slowly, and may be more suitable for determining the effect of water-repellent coatings compared to uncoated samples (Sargent 2019).

Equilibrium humidity tests are the most extensively used test methods involving water vapor (Edvardsen and Sandland 1999; Ohmae et al. 2002; Bekhta and Niemz 2003; Van Acker 2003; Awoyemi 2004; Garcia Estaban et al. 2005; Sik et al. 2010; Ding et al. 2011; Hill et al. 2012; Hill et al. 2013; Rautkari and Hill 2014; Sargent 2019, Humar et al. 2020; Sargent 2022; Bi et al. 2024). ThermoWood® uses the BS EN 318 (2002) European standard for dimensional stability measurements on wood samples (Tourunen 2018). Dynamic vapor sorption measurement is found to be a suitable method for the assessment of the modification degree of wood (Humar et al. 2020). The studies showed that the number of humidity cycles, temperature and humidity conditions appear to be important efficiency on the dimensional stability behavior of wood (Edvardsen and Sandland 1999; Ishimaru et al. 2001; Garcia Estaban et al. 2005; Ding et al. 2011). Rautkari and Hill (2014) suggested that *EMC* change is not a good substitute for directly measuring dimensional stability. The most frequent cause of wood moisture changes in service is changes in RH, and these can happen over extended periods, so it's critical to know how wood will respond to these circumstances (Sargent 2019).

In a previous study by Sargent (2022), furfurylated radiata pine, thermally modified radiata pine and acetylated radiata pine were tested by different methods used for the evaluation of wood dimensional stability. As far as the authors' knowledge, thermally modified ayous and iroko wood are commonly used in Türkiye, and the wood industry is satisfied with the dimensional stability of thermo iroko and ayous wood for outdoor applications, but there is a lack of information in the literature about any standardized or widely agreed-on methods for measuring the dimensional stability of thermally modified iroko and ayous wood. The current study aims to perform such an experimental comparison to understand the differences between results obtained by measuring wood dimensional stability into two basic underlying principles: contacting with liquid water and contacting with various relative humidity until equilibrium is reached. For this purpose, three different test methods (1) repeated water-soaking until saturation, (2) test cycles of water-soaking and drying, and (3) exposure to relative humidity cycles in the air, were selected to measure the dimensional stability of wood samples.

Materials and method

Wood material

Thermo-D class thermally modified iroko (*Milicia excelsa*) and ayous (*Triplochiton scleroxylon*), and their kiln-dried unmodified controls were produced by NovaWood in Bolu, Türkiye and purchased from Perawood Company in Sakarya, Türkiye. The modification procedure is ThermoWood® which is the registered name owned by the Finnish ThemoWood Association. It is based on the controlled modification of timber with heat, steam and water, and it is likely to have been modified at 220 °C for iroko wood and 190 °C for ayous wood. The average tangential swelling and shrinkage due to moisture for Thermo-D class treated wood is 5–6% (ThermoWood Technical sheets 2024). Samples with 2 × 2 × 3 cm (Radial, Tangential, Longitudinal) dimensions were cut from timbers with the initial size of 20 × 25 × 300 cm, and 15 replicates were used for each group. Iroko samples had 15–20

growth rings, while, ayous samples had 3–5 growth rings.

Method-1: repeated water-soaking until saturation

Oven-dried samples were placed into a plastic container filled with deionized water. After 6, 24, 48, 72, 96, 168, 192, 240 and 264 h, the samples were removed from the container and excess water was removed by dabbing with a paper towel. After the dimensions and mass of the samples had been recorded, the samples were again placed in the beaker and the water was replaced. This test was continued for a total of 480 h. Relative water uptake (*WA*), volumetric swelling (*VS*), anti-swelling efficacy (*ASE*), and leaching rate (*LR*) were calculated after each water replacement according to Eqs. 1–4.

$$WA(\%) = \left[\frac{W_2 - W_1}{W_1}\right] \times 100 \quad (1)$$

$$VS(\%) = \left[\frac{V_2 - V_1}{V_1}\right] \times 100 \quad (2)$$

$$ASE(\%) = \left[\frac{VS_{um-VS_m}}{VS_{um}}\right] \times 100 \quad (3)$$

$$LR(\%) = \left[\frac{W_1 - W_f}{W_1}\right] \times 100 \quad (4)$$

where W_2 = wet mass of the samples after each water-soaking period; W_1 = initial dry mass of the samples; V_2 = water-swollen volume of the samples after each water-soaking period; V_1 = initial dry volume of the samples; VS_{um} = volumetric swelling of unmodified samples; VS_m = volumetric swelling of modified samples; W_f = dry mass of the samples after the water-soaking period.

Unmodified (control) samples should be cut from the same material as the modified wood. This gives more accurate results however the timber used in this study came from a range of sources, and using matched control material was impossible. In the study, the average volumetric swelling of the control samples was used for consistency across each water-soaking period.

Method-2: water-soaking and drying-cyclic test

In the water-soaking and drying-cyclic test method (Rowell and Ellis 1978), the mass and dimensions of the oven-dried samples were first recorded and then samples were placed into a plastic container filled with deionized water for 168 h. After 168 h, the samples were removed from the container and excess water was removed by dabbing with a paper towel. After the wet dimensions and mass of the samples had been recorded, the samples were oven-dried, and then the oven-dried dimensions and mass were recorded again. This procedure was named cycle 1 and carried out one more which was named cycle 2 in Fig. 3. Totally 2 cycles of water-soaking and drying performed on the samples. Studied parameters (*WA*, *VS*, *ASE*, and *LR*) were calculated according to Eqs. 1–4 for each water-soaking and drying step. In Fig. 3, Steps 1 and 2 show the parameters in wet and dry conditions after 168 h, respectively, while Steps 3 and 4 show the parameters in wet and dry conditions after 336 h, respectively. As stated above, the average volumetric swelling and shrinking of the control samples were used for consistency across each water-soaking and drying period.

Method-3: relative humidity test

This test was performed according to the principles of BS EN 318 standard (2002) and aims to determine the dimensional changes in samples due to changes in the relative humidity of the air at multiple humidity levels. Normally, this European standard measures dimensional changes in wood-based panels. However, ThermoWood ® uses this standard for dimensional stability measurements on wood samples. Samples were conditioned in three steps (Table 1). After the relative humidity-cycles were completed, samples were oven-dried. Constant mass (less than 0.1% change in mass over 24 h) and dimensions were recorded at each step number, and then volumetric swelling (*VS*) and anti-swelling efficacy (*ASE*) were calculated according to Eqs. 5–10. The average volumetric swelling of the control samples was used for consistency across each humidity level. The equilibrium moisture content (*EMC*) and the moisture excluding efficiency (*MEE*) were defined as Eqs. 11 and 12.

Table 1 Conditioning climates for the wood samples

Step No	Conditioning climates
1	20 °C, 85% relative humidity
2	20 °C, 65% relative humidity
3	20 °C, 30% relative humidity
4	Oven dry until constant mass

$$VS_{d,30} = \left[\frac{V_{30} - V_d}{V_d}\right] \times 100 \quad (5)$$

$$VS_{30,65} = \left[\frac{V_{65} - V_{30}}{V_{30}}\right] \times 100 \quad (6)$$

$$VS_{65,85} = \left[\frac{V_{85} - V_{65}}{V_{65}}\right] \times 100 \quad (7)$$

$$ASE_{d,30} = \left[\frac{VS_{umd,30} - VS_{md,30}}{VS_{umd,30}}\right] \times 100 \quad (8)$$

$$ASE_{30,65} = \left[\frac{VS_{um30,65} - VS_{m30,65}}{VS_{um30,65}}\right] \times 100 \quad (9)$$

$$ASE_{65,85} = \left[\frac{VS_{um65,85} - VS_{m65,85}}{VS_{um65,85}}\right] \times 100 \quad (10)$$

$$EMC_{d,85} = \left[\frac{W_{85} - W_d}{W_d}\right] \times 100 \quad (11)$$

$$MEE = \left[\frac{EMC_{umd,85} - EMC_{md,85}}{EMC_{umd,85}}\right] \times 100 \quad (12)$$

where $VS_{d,30}$ = volumetric swelling for a change of the relative humidity from 0 to 30%, in percent; $VS_{30,65}$ = volumetric swelling for a change of the relative humidity from 30 to 65%, in percent; $VS_{65,85}$ = volumetric swelling for a change of the relative humidity from 65 to 85%, in percent; V_d = dry volume of the samples, V_{30} = volume at 30% relative humidity; V_{65} = volume at 65% relative humidity; V_{85} = volume at 85% relative humidity; VS_{um} = volumetric swelling of unmodified samples; VS_m = volumetric swelling of modified samples; W_{85} = mass of the samples at the 85% relative humidity; W_d = dry mass of the samples; EMC_{um} = EMC of unmodified samples, in percent; EMC_m = EMC of modified samples, in percent.

Color and FTIR analysis

Twenty color measurements were taken from the tangential surface of modified and control samples (Konica Minolta CM2600D, Ramsey, NJ, USA) by determining the *CIE L*a*b** color system. Measurements were carried out with a sensor of 8 mm diameter and an observation angle of 10°. Change in color coordinates (ΔL^*, Δa^*, Δb^*) and total color change (ΔE^*) were calculated for the samples to determine the effect of leachates on wood color in the water-soaking test method (1) and cyclic water soaking and drying test method (2) where contact with liquid water was involved. FTIR analysis of thermally modified samples was performed with the Bruker Optics Tensor 37 device (Bruker, Karlsruhe, Germany). FTIR spectra were recorded from the wood surface with an ATR unit by scanning at least 2 points on the same surface. The wavenumber range was 400–4000 cm^{-1} and the resolution was 4 cm^{-1} as the measurement settings. Spectra were baseline-corrected and smoothed by the OPUS software.

Statistical analysis

The color measurement (ΔL^* and ΔE^*), *ASE,* and *EMC* results were analyzed by the simple and multivariate analysis of variance (ANOVA) in IBM SPSS 22 statistical software, and test methods were classified by Duncan groups ($p < 0.05$). *ASE* values of samples after 480 h in the water-soaking test method (Method-1), *ASE* values in Step 4 of cyclic water soaking and drying test method (Method-2), and $ASE_{65,85}$ values in the relative humidity test (Method-3) were used for statistical analysis. *MEE* values of samples were analyzed by Independent Samples T-Test.

Results and discussion

Chemical proof of thermal modification

Figure 1 shows the FTIR spectra of thermally modified wood and control wood. The FTIR spectra of thermally modified and unmodified iroko and ayous wood reveal significant structural changes, particularly in the hemicellulose-rich region (1730–1740 cm^{-1}). Thermal modification reduces hemicellulose content, evidenced by the diminished peak intensity at this wavelength. This finding aligns with prior research demonstrating that hemicellulose, being the least thermally stable wood component, degrades significantly under heat treatment, producing acetic acid and other by-products that catalyze further decomposition (Kubovský et al. 2020). On the other hand, chemical changes more prominently can be observed in modified iroko spectrum than modified ayous. This is more likely due to the extractive content (Altgen et al. 2016). Additionally, the spectra indicate alterations in the cellulose structure, especially amorphous regions (1160–900 cm^{-1}), with shifts and peak sharpening suggesting partial depolymerization. This transformation contributes to the enhanced dimensional stability and reduced hygroscopicity of thermally modified wood, corroborating findings in earlier studies (Esteves and Pereira 2009). Peaks associated with lignin (1500–1600 cm^{-1}) exhibit subtle changes in intensity, likely due to the relative concentration of lignin as hemicellulose and cellulose diminish. The absorption peak at 2925 cm^{-1} in the FTIR spectrum represents the stretching vibrations of C–H in methyl and methylene groups. This region provides insight into the degradation of polysaccharides within hemicelluloses during thermal treatment. As hemicelluloses undergo thermal decomposition, the number of methyl and methylene groups decreases, leading to diminished peak intensity (Liang and Wang 2017). Particular attention should be given to the spectral regions highlighted by pink zones, as these correspond to molecular vibrations related to C=O stretching in hemicelluloses and aromatic skeletal vibrations in lignin. These areas emphasize the complex interplay between degradation and structural reorganization during thermal modification. Enhanced aromaticity and reduced polar groups contribute to the improved resistance of the wood to biological degradation and moisture absorption.

Finally, the comparative analysis of iroko and ayous samples suggests species-specific responses to thermal modification. Variations in lignin composition and hemicellulose type, as well as extractives of

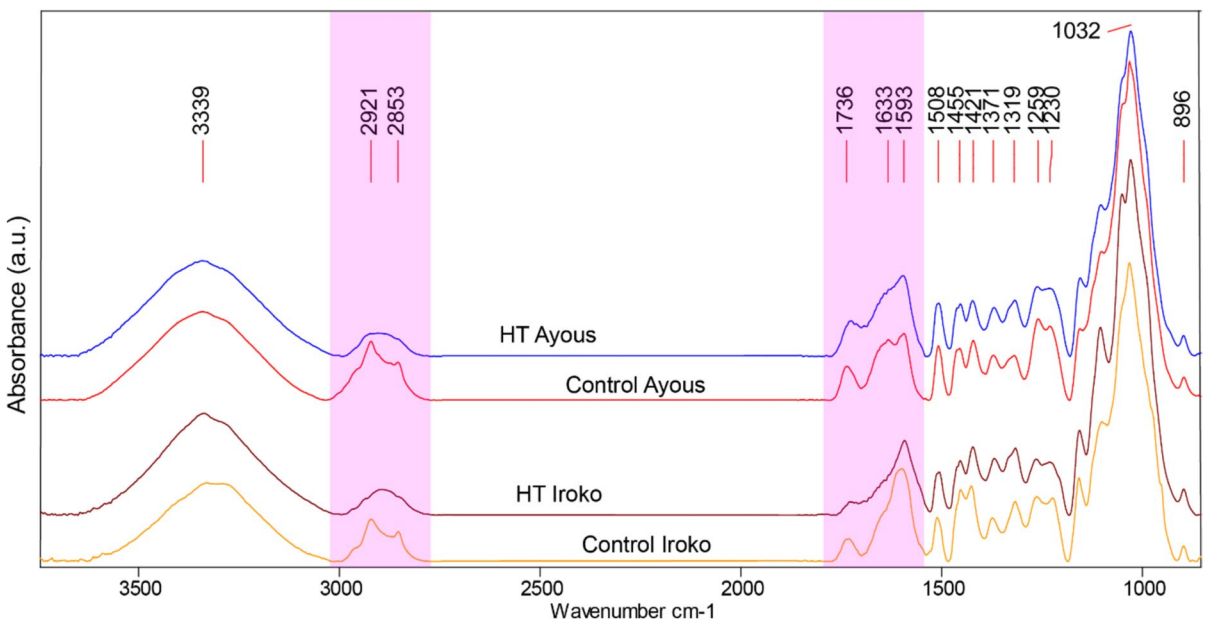


Fig. 1 FTIR spectra of thermally modified wood and control wood (HT: Thermal modification; Control: Unmodified)

iroko likely influence the thermal degradation pathways, as observed in the distinct intensity changes across the spectra. Such differences highlight the importance of tailoring thermal treatment parameters to the specific chemical composition of the wood species for optimal results (Gaff et al. 2023).

Method-1: repeated water-soaking until saturation

Water uptake (%) of iroko and ayous samples was continually increased along with water-soaking periods (Fig. 2). Water uptake levels in samples were much more pronounced during the early part of water-soaking periods, especially within 48 h. After 168 h of water-soaking period, water uptakes slightly increased due to high water uptake into available empty pores or voids in wood at the beginning of soaking and the reduction of those wood spaces over time (Baysal et al. 2006; Tomak et al. 2011). The water uptakes of thermally modified samples and iroko samples were slightly lower than that of the controls and ayous samples during both the early and later stages of the test. Lower uptakes by iroko samples in comparison to ayous samples were due to the anatomy of ayous which is a low-density wood species that has more empty pores or voids in its structure where water fills in. High-density wood species have proportionately more cell walls and fewer voids than low-density wood species (Dias et al. 2019).

Volumetric swelling (%) and anti-swelling efficiency (%) were also more pronounced during the first 48 h of water-soaking periods (Fig. 2). The results demonstrated that thermal modification in ayous wood ensured a relatively lower volumetric swelling rate in comparison to control samples whilst the improvement in volumetric swelling was remarkably less in thermally modified iroko samples compared with the controls. Consistent with the results of volumetric swelling rates, anti-swelling efficiency which was the reduction of volumetric swelling in percentage between the thermally modified and control samples, was found to be greater in thermally modified ayous samples (~45%) than in thermally modified iroko samples (~25%). The anti-swelling efficiency results also showed that better linear

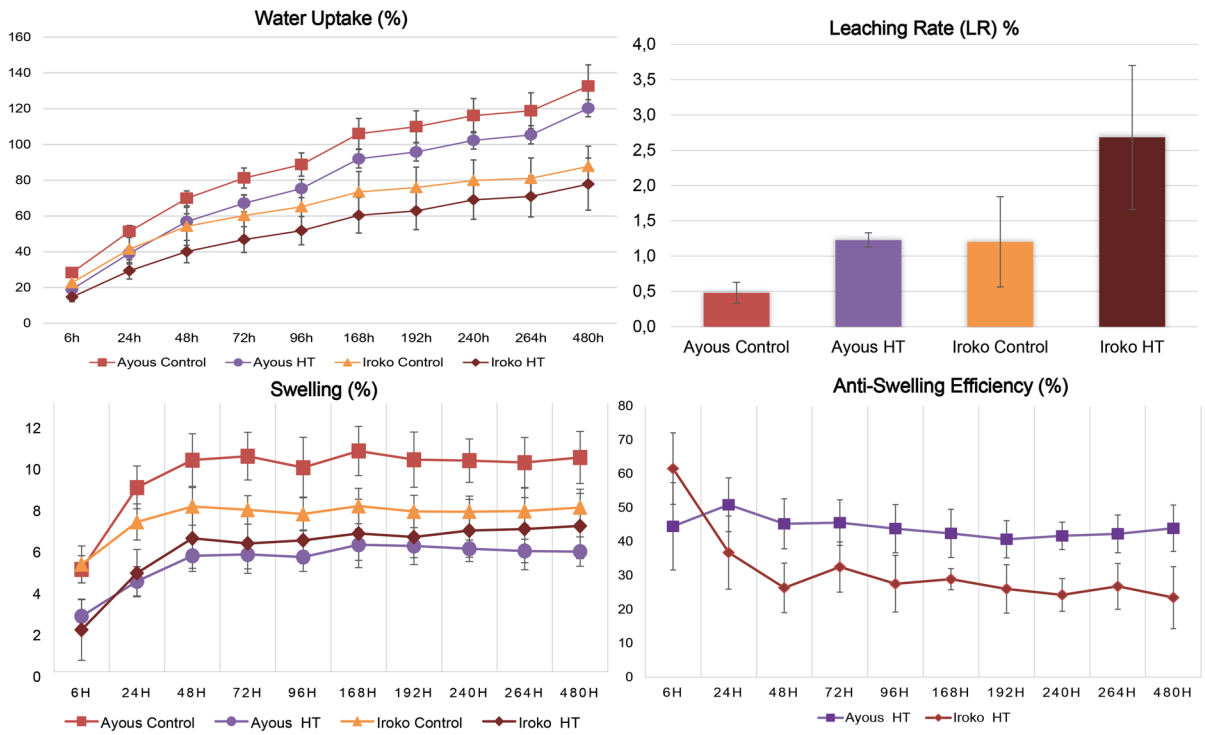


Fig. 2 Water uptake (%), swelling (%), anti-swelling efficiency (%), and leaching rate (%) of samples during the water-soaking test

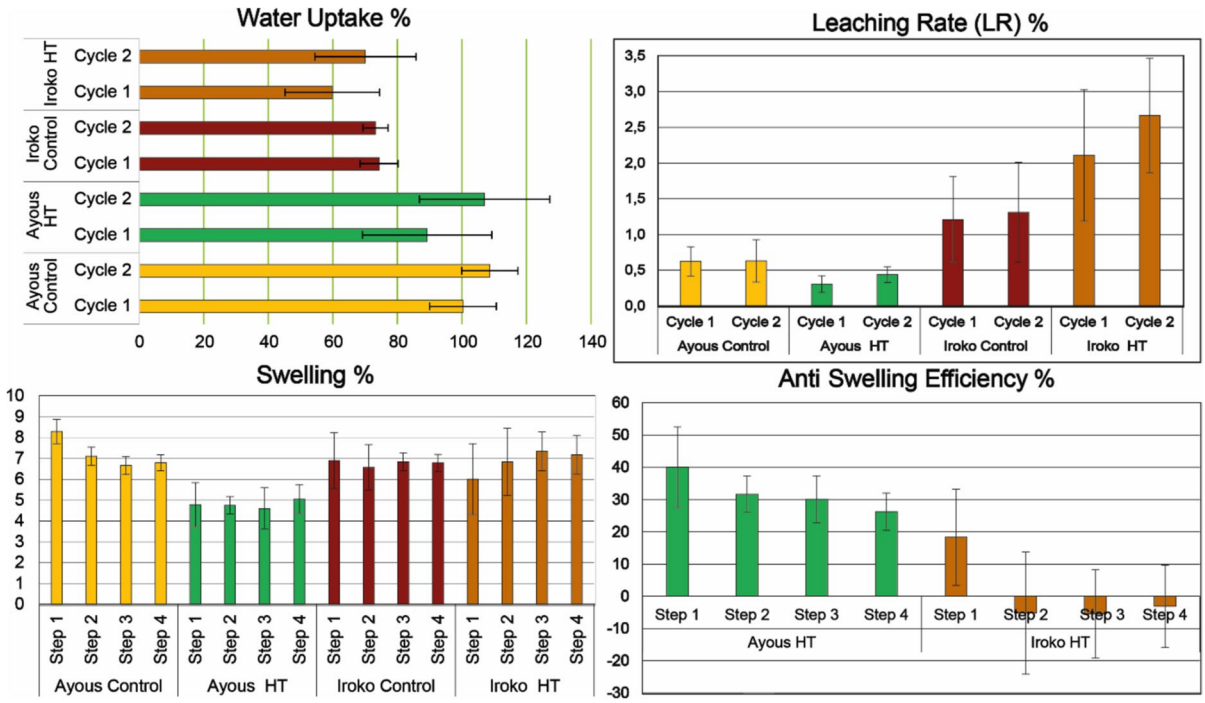


Fig. 3 Water uptake (%), swelling (%), anti-swelling efficiency (%), and leaching rate (%) of samples during wetting and drying cycles

stability was achieved in ayous wood samples during the water-soaking period, whilst in iroko samples this stability showed less linearity throughout the process.

Leaching rates during the water-soaking test were 2.68% for thermally modified iroko and 1.2% for control iroko samples. In the case of ayous wood, thermally modified and control samples had 1.23% and 0.48% leaching rates, respectively (Fig. 2). For both wood species, thermal modification caused more than two times the leaching rate in samples in comparison to control samples. Water-leachable components or degradation products on the surface after the thermal modification process were the reasons for the higher leaching rates in thermally modified samples. The leaching of thermal breakdown products of the wood cell wall in thermally modified wood was reported by Altgen et al. (2018).

The possible explanation for the above-mentioned results both in *ASE* and leaching rate evaluations would be the diversity in extractives between the wood species, and also, differences in the wood components. Ayous wood possesses a heartwood that is much less differentiated from sapwood and contains few extractives (Tchinda et al. 2018). Biwole et al. (2022) identified some polyphenol oligomers in the stilbene class such as geranyl trihydroxy stilbene and chlorophorin which are highly hydrophobic substances, in iroko, and its known that high concentrations of polyphenols in the extractives of iroko wood are sufficient to provide water resistance, and water resistance was closely related to the type of extractives and not to the amount of extractives. Thermal degradation of hydrophobic extractives in iroko wood during high temperatures and leaching of that degraded extractives and/or wood components from wood samples throughout the water-soaking test might cause lower dimensional stability in iroko wood which is known as extractive-rich wood specie than ayous known as non-durable wood specie. Gaff et al. (2023) well-reviewed the extractive content change of iroko wood during the thermal modification process such that it first increases and then decreases. They reported that extractive composition changes at higher temperatures—some disappear, and new ones are formed, and the extractives can migrate to the surface during thermal modification. The decrease in

extractive content is because of their removal from the wood or degradation during the thermal modification process (Gaff et al. 2023). Most of the raw extractives decreased and new compounds including anhydrosugars, mannosan, galactosan, levoglucosan, and two C5 anhydrosugars were produced, besides large amounts of decomposition products of lignin such as syringic acid, sinapaldehyde, and syringaldehyde according to Esteves et al. (2011) and Candelier et al. (2020). The extractive content of wood increased by thermal modification at low temperatures but decreased with modification at high temperatures. At high temperatures, new compounds were generated, but the heat converted them into volatile products, which resulted in a decrease in extractives (Wang et al. 2016; Candelier et al. 2020; Gaff et al. 2023). Similar statements were also reported by Hill et al. (2021) that fats and waxes migrate along flow pathways, such as ray parenchyma at temperatures as low as 100 ℃, and most of the native extractives are progressively lost or degraded as the process temperature increases. The 1593 cm^{-1} band intensity decreased in the spectra of thermally modified iroko wood (Fig. 1). It is known that this band originates from phenolic ring vibrations, and it was also concluded that some of the phenolic groups, which are known to be present in the structure of many extractives, were degraded because of the thermal treatment.

Method-2: water-soaking and drying cycles

The water uptake of ayous samples was higher than iroko samples in this method similar to previous findings in the water-soaking test (Method-1). Prolonged water-soaking periods caused more water uptakes which were more pronounced for ayous samples than iroko samples (Fig. 3). The effect of the thermal modification in reducing water uptake levels in modified wood was much more pronounced in Cycle 1 in comparison to controls for both wood species, however, only a slight decrease is observed in modified samples compared to controls in Cycle 2. The overall findings in water uptakes showed thermal modification did not prevent or decrease water uptake of ayous and iroko wood in long periods of water-soaking for 336 h.

The improvement in dimensional stability by thermal modification of ayous wood can be proved by the decrease of volumetric swelling compared to control ayous wood, and therefore by the increase in anti-swelling efficiency rates (Fig. 3). The trend was close to but somehow different from that of volumetric swelling and *ASE* of modified ayous wood in the previous method (Method-1). Two important differences stand out in modified ayous wood. The first was *ASE* of modified ayous wood decreased from Cycle 1 to Cycle 4 in this method, whilst it was very stable during all water-soaking periods in Method-1, and the second was an average rate of *ASE* of ~ 30% in this method. It was ~ 33% lower than the *ASE* found in Method-1 for modified ayous wood. Wetting and drying cycles in Method-2 caused more degradation in wood. They could negatively affect polymer network and crosslinking within the cell wall polymers which are previously gained in wood with the thermal modification process. Although no visible macro cracks occurred on the modified ayous wood, some nonvisible micro-cracks in wood might decrease the *ASE* of samples in prolonged wetting and drying cycles (Fig. 4). Although no direct evidence of cracking affects *ASE* measurements (Sargent 2019), cracking due to drying stress in wet samples could reduce *ASE* levels in this study. Another reason for decreasing *ASE* would be the further leaching of degraded extractives and components in each step, drying but especially second water-soaking. Thermally modified iroko wood ensured ~ 18% dimensional stability in the first cycle of the wetting step (Step 1) while in other steps from Step 2 to Step 4, no further improvement in dimensional stability was observed since control iroko wood swelled less than modified iroko. Visible macro cracks shown with red arrows in Fig. 4 were developed in the drying step of the second cycle (Step 4), and probably, some micro-cracks had already developed in the drying step of the first cycle (Step 2) and these were gradually deepened and increased in number after Step 4 (Fig. 4). Those cracks might occur since the wet samples were not gradually dried. Here, it is noteworthy that modified samples exposed to the same treatment steps as controls formed more cracks than control samples. Cracks could weaken the anti-swelling efficiency of wood. It should be also noted that wood defects occurring during thermal modification such as cracking, breaking cell walls and a collapse of vessels in wood also influence the results (Boonstra et al. 2006; Bastani et al. 2015). Thermally modified wood is more susceptible to crack formation and crack propagation (Majano-Majano et al. 2012).

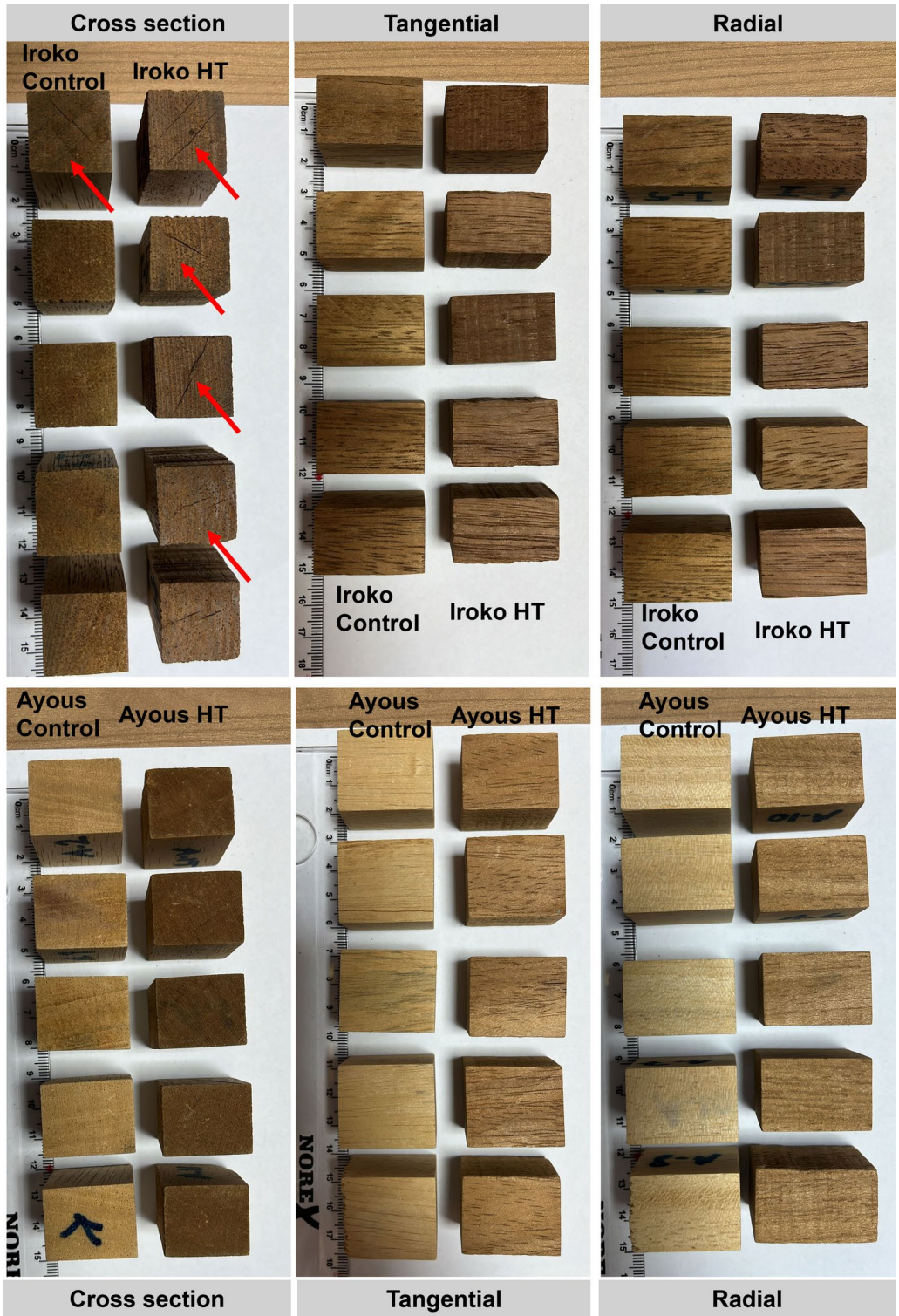


Fig. 4 Cross sections of wood samples after the test **a** Ayous control and ayous heat treated wood, **b** Iroko control and iroko heat treated wood

Severe stresses during the ThermoWood® process are likely to occur due to the following reasons (1) the high-temperature drying step, which decreases the moisture content to nearly 0% before the actual thermal modification step, leading to a maximum shrinkage of the wood, or (2) the final conditioning step, where water is sprayed on the wood, creating a fast swelling of the wood at the surface (Syrjanen and Kangas 2000; Altgen et al. 2015). Altgen et al. (2015) observed in microscopic analysis that the parenchyma and epithelial cells within the rays are frequently damaged during the thermal modification process. These micro-defects might act as a starting point for more severe cracks and thus lead to a higher sensitivity towards cracking during repeated swelling and shrinkage of wood (Altgen et al. 2015). High-density wood species such as iroko wood are more prone to cracking than low-density wood species such as ayous wood since higher-density wood species swell and shrink at a greater rate (Dias et al. 2019). Sargent (2022) stated the reason for the difference in swelling behavior between the first, and subsequent soaking cycles is the relaxation of any stresses in the wood, and the effect of any differences in drying history, which is no longer present after the first soaking cycle. Altgen et al. (2018) attributed the difference in swelling behavior between the first, and subsequent soaking cycles to the leaching of compounds from the sample during the first water-soaking, and this affects the stability in subsequent soaking cycles. This leaching could be from thermal breakdown products of the wood cell wall (Altgen et al. 2018).

Leaching rates in Cycle 1 and Cycle 2 during the test were 2.11% and 2.66% for thermally modified iroko, and 1.21% and 1.31% for control iroko wood, respectively. Thermal modification increased the leaching rates in iroko wood due to two following reasons (1) leaching of thermal breakdown products such as degraded extractive products, and (2) more crack formations in modified samples than controls. Ayous wood behaved somehow differently from iroko wood. The leaching rates were 0.33% and 0.44% for thermally modified ayous wood at two cycles, respectively, and 0.63% for control ayous wood for both cycles. On the other hand, control ayous wood had a greater leaching rate than thermally modified wood which can be related to the starch content in ayous wood. Ayous is known as one of the starch-rich wood species, and starch in control wood can become more leachable in wetting and drying cycles. During the water leaching treatment, starch and sugar were removed through dissolution and leaching, and a higher number of available voids for water to occupy (do Amaral et al. 2023). Pectin and starch become hydrolysed at around 200 °C (Sundqvist 2004), and therefore the leaching rates in water were found to be lower in modified samples than controls.

Color measurement results also confirmed the leaching rates and claims on *ASE* findings in the water-soaking test (Method-1) and cyclic water-soaking and drying test (Method-2). The color measurement results and their statistical test (Duncan homogeneity groups in simple variance analysis) results on ΔL^* and ΔE^* values for each wood species are given separately in Table 2. As can be seen from ΔL^* values of ayous and iroko wood, brightening was observed in both modified samples and control samples after water-soaking tests. This could be due to leaching the degraded components from the modified samples, and the leaching of water leachable components from the controls. The dimensional stability test methods statistically affected ΔL^* and ΔE^* values for ayous wood ($p < 0.05$). Method-1 caused more brightening in modified ayous samples than Method-2 since wood samples were submerged longer in water in Method-1 than in Method-2. In the case of modified iroko samples, Method-1 and Method-2 had a similar effect on wood color. Although water submersion time was longer in Method-1 than in Method-2, deep cracks formed in Method-2 could increase the leaching of components which brightened the color. Multiple variance analysis was also performed on ΔL^* and ΔE^* values to compare the dimensional stability tests and wood species. The results were found to be significant ($p < 0.05$). Regardless of the dimensional stability tests, more color change was observed in iroko wood compared to ayous wood. In terms of dimensional stability tests, the darkest surfaces were found as follows: initial surfaces before dimensional stability tests > Cyclic water soaking and drying test (Method-2) > Water-soaking test (Method-1).

Method-3: relative humidity test

The equilibrium moisture content (%), moisture excluding efficiency (%), swelling (%) and anti-swelling efficiency (%) of samples under different

Table 2 Color change of samples after thermal modification and water-contact dimensional stability tests

Groups		ΔL^*	Δa^*	Δb^*	ΔE^*
Thermal modification	Ayous HT	− 35.96 (0.56) E*	6.44 (0.18)	− 1.99 (0.54)	36.59 (0.55) A
Water-soaking test (Method-1)	Ayous HT	7.76 (1.56) A	− 0.12 (0.15)	3.80 (0.67)	8.65 (1.66) C
	Ayous Control	− 4.26 (0.99) C	0.18 (0.36)	1.88 (0.66)	4.70 (1.09) E
Cyclic water soaking and drying test (Method-2)	Ayous HT	5.57 (2.60) B	0.32 (0.55)	2.73 (0.77)	6.32 (2.50) D
	Ayous Control	− 12.08 (3.19) D	2.56 (0.82)	4.17 (1.11)	13.09 (3.24) B
Thermal modification	Iroko HT	− 32.93 (1.44) C	3.25 (0.60)	− 10.27 (1.18)	34.66 (1.61) A
Water-soaking test (Method-1)	Iroko HT	7.05 (2.39) A	0.52 (0.31)	3.97 (1.61)	8.13 (2.84) C
	Iroko Control	− 19.43 (1.11) B	2.39 (0.81)	− 0.44 (1.50)	19.66 (1.16) B
Cyclic water soaking and drying test (Method-2)	Iroko HT	6.41 (3.01) A	1.03 (0.36)	4.54 (2.38)	7.95 (3.80) C
	Iroko Control	− 19.92 (2.48) B	2.94 (0.61)	− 1.35 (1.66)	20.24 (2.62) B

*Letters indicate homogeneous groups according to Duncan homogeneity groups in statistical analysis (ANOVA, $p < 0.05$)

relative humidity conditions (30%, 65% and 85% RH) are shown in Fig. 5. The decrease in *EMC* after wood modification may serve as a general indication of enhanced dimensional stability, indicating that the interaction between water and wood has been changed (Sargent 2022). *EMC* of modified iroko samples was 8% lower than that of modified ayous samples at 85% RH. In the case of control samples, iroko samples had 17% lower *EMC* than ayous samples due to iroko has less empty pores or voids in its structure where less water vapour absorption occurs, and high concentrations of polyphenols in the extractives of iroko wood which provide water resistance (Biwole et al. 2022). *EMC* values of samples were analyzed by simple variance analysis and significant differences were found ($p < 0.05$). Statistical order in *EMC* values according to Duncan homogeneity groups was found as follows: Ayous control > Iroko control > Modified

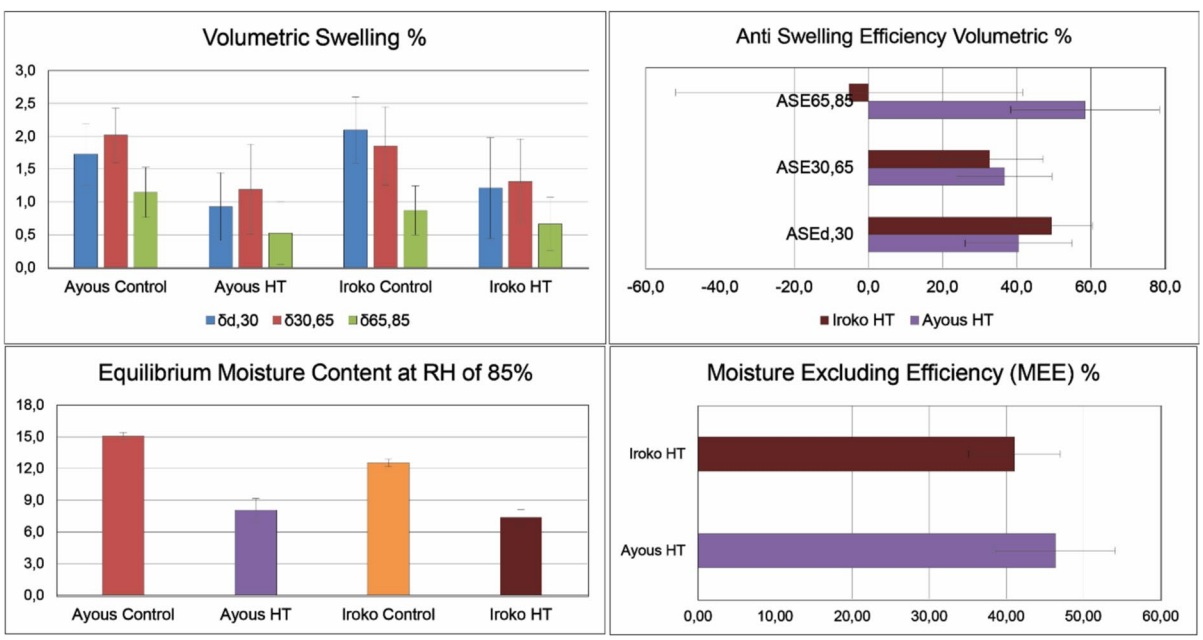


Fig. 5 The equilibrium moisture content (%), moisture excluding efficiency (%), swelling (%) and anti-swelling efficiency (%) of samples under different air humidity conditions

ayous > Modified iroko. *MEE* was found to be 41% for modified iroko samples and 46% for modified ayous samples. *MEE* values were analyzed by the independent samples T-Test, and significant difference was not found between each group ($p > 0.05$).

Thermally modified ayous wood had lower volumetric swelling than control wood after exposure to all humidity conditions and had *ASE* between ~ 40% to ~ 60%. *ASE* of thermally modified ayous samples during Steps 1 and 2 was found as ~ 60% whilst it was found as ~ 40% during Steps 2–4 (Table 1). The findings show that thermally modified ayous wood had ~ 33% greater dimensional stability in higher humidity conditions than in lower humidity conditions. Iroko wood behaved somehow opposite trend from ayous wood. Dimensional stability was not observed since control iroko wood swelled less than modified iroko wood during Steps 1 and 2 (from 85 to 65%), however, ~ 30% and ~ 50% *ASE* were calculated during Steps 2 and 3 (from 65 to 30%), and 3 and 4 (from 30% to oven dry), respectively. These findings clearly showed that thermally modified iroko wood did not ensure any dimensional stability in higher humidity conditions whilst the modified wood had ~ 30–50% *ASE* in lower humidity conditions. This observation is consistent with the results found on thermally modified iroko wood in Method-2 which shows thermally modified iroko wood can not ensure dimensional stability under high humidity conditions (e.g. 85% RH) and prolonged wetting and drying cycles. Such an observation can be explained by the degradation of the high extractive content of iroko wood which lost its hydrophobicity due to thermal degradation and removal from the cell walls at high humidity and saturated conditions.

Dimensional stability test results were analyzed with multiple variance analyses in terms of three methods and two wood species. Accordingly, it was determined that the methods and wood species had a statistical effect on dimensional stability values ($p < 0.05$). Ayous wood had a higher *ASE* value compared to iroko when all methods were considered. *ASE* values calculated from different dimensional stability methods were found in the following order according to Duncan homogeneity groups: Water-soaking test (Method-1) > Relative humidity test (Method-3) = Cyclic water soaking and drying test (Method-2).

Conclusions

In the water-soaking test (Method-1) and cyclic water-soaking and drying test (Method-2) where contact with liquid water was involved, thermally modified iroko wood absorbed less water than thermally modified ayous wood. However in the case of dimensional stability measurements, modified ayous wood showed more dimensional stability than modified iroko wood in both methods. In fact, in Method-2 with wetting–drying cycles, dimensional stability was not obtained in modified iroko wood as the exposure time was prolonged, and deep cracks were observed on the cross-section of the samples. The cracks were more in modified iroko samples than in the controls, whilst modified ayous wood had no visible cracks. In method-3, which involves contact with water vapor until equilibrium is reached, modified ayous wood showed higher dimensional stability at higher relative humidity (85% RH), while iroko wood showed the opposite behavior. The results of the three different methods clearly showed that thermal modification ensured good dimensional stability for ayous wood, whereas in iroko wood, dimensional stability decreased as the prolonged water immersion period (Method-1) and in some cases could not be achieved (Methods 2 and 3) as the wetting–drying cycles increased and keeping wood in high relative humidity such as 85%. As has been long known, thermal modification decreases the mechanical strength of wood. Considering the strength losses besides the unpleasant results of dimensional stability measurements in this study, the necessity of thermal modification in iroko is a controversial issue. When the three methods are evaluated in terms of their practical applicability, Method-3 seems not to be a practical experiment since the method requires a long time and special climate cabinets. Long-term wetting and drying-cyclic test (Method-2) is recommended for dimensional stability evaluations in modification studies since wood material tested with the harshest conditions in the laboratory may better mimic real outdoor exposure conditions. Thermal modification of extractive-rich wood species should be extensively investigated in terms of dimensional stability measurements with long-term wetting and drying-cyclical tests since the treatment itself may not be suitable for these species like as non-durable wood species. FTIR spectroscopy

also confirms the decomposition of iroko extractives which supports dimensional stability results.

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Author contribution EDT: Conceptualization, methodology, investigation, data curation, visualization, writing—original draft MAE: Conceptualization, methodology, data curation, visualization, writing—review & editing.

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Data availability Data sets generated and/or analyzed during the current study are available from the corresponding author upon request.

Declarations

Conflicts of interest The authors declare no competing interests.

Ethical approval Not applicable.

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