Physical-Mechanical Properties of Heartwood and Sapwood in *Toona sp.* Wood (*Toona ciliata* M. Roem.) Before and After Accelerated Aging Treatment

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The differences of physical-mechanical properties in heartwood and sapwood of *Toona sp.* wood (*Toona ciliata* M. Roem.) were compared before and after an accelerated aging treatment, which was conducted in accordance with ASTM (American Society for Testing and Materials) standards. The results revealed that the physical-mechanical properties of heartwood were better than those of sapwood. The accelerated aging treatment weakened the physical-mechanical properties of the wood samples. During the aging treatment, the carbohydrates in the cell wall, including cellulose and hemicellulose, were depolymerized; as such, the crystallinity of cellulose was decreased by aging. The depolymerization of carbohydrates and the reduction of crystalline contributed to decreasing the physical-mechanical properties of the wood samples.

Keywords: Accelerated aging treatment; Heartwood and sapwood; Wood properties; Wood chemicals

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INTRODUCTION

Toona sp. wood (*Toona ciliata* M. Roem.) is an important fast-growing commercial wood species in China. It has been widely used in the furniture industry and interior decoration due to its beautiful reddish-brown heartwood surface color.

When sapwood is transformed to heartwood, the starch in the parenchyma cells disappears, and the parenchyma cells are lignified as a result (Bergström 2003). These changes cause variation in the physical-mechanical properties between sapwood and heartwood. For instance, the permeability of heartwood will be lower than that of sapwood, which is caused by the obstruction of transportation cells (Taylor *et al.* 2002). However, the density, mechanical strength, and natural durability of heartwood is greatly improved (Long *et al.* 1981). These differences between heartwood and sapwood allow them to be utilized in different ways.

The physical-mechanical properties of woody materials tend to decrease with age, which has a negative effect on their service life. Therefore, it is crucial to evaluate the influence of the aging process on wood properties. In general, there are two methods to evaluate the changes of wood with aging: long-term testing (outdoor exposure test) and short-term testing (accelerated aging test). The deterioration of the bending properties after the 5 years' long-term testing is the same as that of the short-term treatment (Kojima and Suzuki 2011). The latter often is more appropriate than the outdoor exposure test, as it can be completed in a short period of time in an indoor space (Huang *et al.* 2014).

Wood is composed of three main molecules: cellulose, hemicellulose, and lignin.

These molecules play an important role in determining the physical-mechanical properties of wood. Hence, it is important to evaluate the variation of the main chemical components in the wood by applying Fourier transform infrared spectroscopy (FT-IR). Moreover, the aging process of wood may lead to a change in crystallinity, which also influences the elasticity, absorptive capacity, and other valuable physical-mechanical properties of wood (Bhuiyan *et al.* 2000). The changes in the crystallinity of wood are evaluated by X-ray photoelectron spectroscopy.

To date, there has been no study on physical-mechanical properties of *Toona* sp. wood before and after aging treatment. In this work, the specific objective is to determine the physical-mechanical properties of sapwood and heartwood in *Toona* sp. wood before and after aging.

EXPERIMENTAL

Materials

Toona sp. trunks (53 years old) were harvested from Dechang, Sichuan, China, in 2014. Heartwood and sapwood zones were identified through visual inspection. As shown in Fig. 1, section A was used to study anatomical characteristics. Samples for physical-mechanical properties were taken from section B.

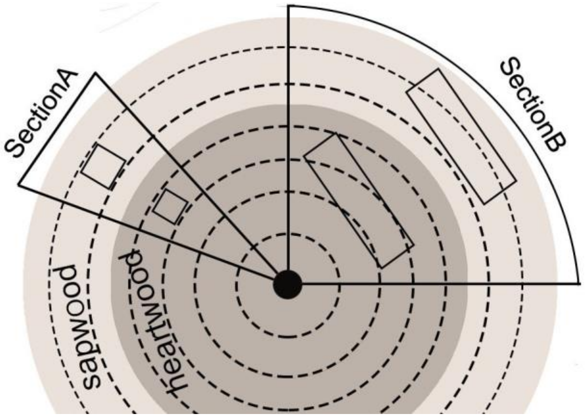

Fig. 1. Transversal section of the *Toona* sp. wood

Methods

Anatomical characteristics

Samples with size dimensions of 5 mm × 5 mm × 5 mm were boiled in distilled water until soft, then sliced into sections with a 30 µm thickness using a sliding microtome (TU-213, Yamato Kohki Instruments Co., Ltd, Asaka-shi, Japan). Wood slices were stained with a 0.1% Safranin O (Alfa Aesar, Ward Hill, MA, USA) for 30 s to increase visibility and then dehydrated by an alcohol series (30, 50, 70, 90, 95 and 100%) for 10 min each. One drop of neutral balsam (Shanghai Specimen and Model Factory, Shanghai, China) was added to the center of the slide. The cross-section was then mounted on the slide and covered with a coverslip. Air-dried slides were observed with a digital camera microscope (BX51, Olympus, Tokyo, Japan) and analyzed with a wood anatomical analysis software (STDY-M, Tiandiyu Technology, Beijing, China).

The proportion of axial parenchyma, fiber, ray, and vessel was measured according to the method outlined by Pirralho *et al.* (2014) on the transverse section using a 70 point-grid on successive areas along the radius.

To measure the dimensions of the fiber and vessel, wood samples were splintered into match stick size. Wood cells were then isolated using Jeffrey's solution (10% chromic acid: 10% nitric acid mixtures = 1:1). Each of the fifty complete and reasonable fiber and vessels were randomly selected and measured for each part. The measurement was carried out using a digital camera microscope.

Accelerated aging treatment

The accelerated aging treatment was carried out in accordance with the standard method ASTM (American Society for Testing and Materials) D 1037-99 (1993). The procedure consists of 6 cycles of the following sequence: soaking in water for 1 h at 50 °C, steaming at 95 °C for 3 h, and freezing at -12 °C for 20 h; drying at 100 °C for 3 h, steaming at 95 °C for 3 h, and drying at 100 °C for 18 h. In total, 12 days were required. After the accelerated aging procedure, the samples were conditioned at 22 °C and 65% relative humidity for 6 weeks prior to testing.

Chemical analysis

Wood powders that passed through a 40 mesh sieve and were retained on a 60 mesh sieve were dried at 80 °C to a constant weight. The hemicellulose, alpha-cellulose, Klason lignin content, ethanol-toluene extracts, 1% NaOH solubility, and ash contents of these samples were determined according to ASTM standards D1104-56 (1971), D1103-60 (1971), D1106-96 (1996), D1107-96 (1996), D1109-84 (2001), and D1102-84 (2001), respectively. Hemicellulose content was determined as described in Zhang *et al*. (2011).

Determination of physical-mechanical properties

The physical properties of the wood samples were determined according to GB (National Standard of the People's Republic of China)/T 1933 (2009) and GB/T 1932 (2009). The basic density (*D*, g/cm^{-3}) was determined based on the measured oven-dry weight and green volume. Volumetric shrinkage (*S*, %) was estimated with air and oven dry samples. The samples for basic density and volumetric shrinkage were oven dried at 103 ± 2 °C until a constant weight was obtained. The volume of the sample was determined using a water displacement method.

The mechanical properties, *i.e.*, compressive strength (CS, MPa), modulus of rupture (MOR, MPa), and modulus of elasticity (MOE, GPa), and hardness (KN) of the cross-sections were determined according to GB/T 1935 (2009), GB/ T1936.1 (2009), GB/T 1936.2 (2009), and GB/T 1941 (2009), respectively, using a universal testing machine (model RGM-4100, Reger, Shenzheng, China).

CS was measured by loading the specimen at a constant rate of 0.5 mm/min until the maximum load was reached or failure occurred. The bending tests were conducted under a three-point bending device at the cross head vertical speed of 10 mm/min. A free span length of 240 mm was required for the MOR and MOE tests. Hardness was measured by pressing the hemispherical steel into the sample at a constant rate of 5 mm/min until the depth was 5.64 mm. Thirty replicates were carried out for each group.

FT-IR spectroscopy

The FT-IR analysis was performed with a Nicolet Nexus 670 spectrometer from Madison Instruments (Middleton, WI, USA) equipped with a Thermo Nicolet Golden Gate MKII Single Reflection ATR accessory equipped with a hybrid diamond/Znse single crystal (Brilliant Spectroscopy, Radnor, PA, USA). A small quantity of sample powders was covered, flatwise, on the detection window. Each sample was analyzed in the range of resolution from 400 cm^{-1} to 4000 cm^{-1} with a spectral resolution of 4 cm^{-1}, and 32 scans were collected. Three replicates were conducted in this work. The spectra were normalized at the band at 1506 cm^{-1}.

X-ray measurements

The wood sample was treated using an X-ray diffractometer (Philips X-Pert, Panalytical, Almelo, Netherlands) with an X-ray generator and a Cu target (λ=0.1541 nm): with a Kα (40 kv, 40 mA) radiation at room temperature and a scan rate at 2.5 ^{o}/min. The intensity was measured as a function of the scattering angle of the 2θ by θ-2θ scan. The sample was spread over a range of $2\theta = 8$ to 40° (Bhuiyan *et al*. 2001; Zhang *et al*. 2011). The degree of crystallinity (C_rI) was determined by the Segal method (Li 2003) (Eq. 1),

$$C_r I = \frac{(I_{002} - I_{am})}{I_{002}} \times 100\% \qquad (1)$$

where I_{002} is the diffraction intensity of the crystallizing area from the (002) plane at 2θ = 22.2°; I_{am} is the intensity of the amorphous background scatter measured at 2θ = 18°. I_{002} represents both crystalline and amorphous material, and I_{am} represents only the amorphous material (Inagaki *et al.* 2010).

Statistical analysis

Statistical analysis was carried out using SAS (version 9.1, SAS Institute, Cary, NC). The mean and the standard deviation were calculated.

RESULTS AND DISCUSSION

Anatomical Features between Heartwood and Sapwood

Table 1 shows the mean value of fiber morphologies in heartwood and sapwood. The fiber length in sapwood (1.15 mm) was longer than that of heartwood (1.01 mm). This observation was also noted by Li *et al*. (2014), suggesting that the maturing process of fiber varies from heartwood to sapwood.

Table 1. Anatomical Features of Fiber of *Toona* sp. Wood

Zone	Length (mm)	Width (μm)	L/W	Lumen Diameter (μm)	Double Cell Wall Thickness (μm)
Heartwood	1.01 ± 0.14	22.45 ± 2.51	45.00 ± 3.21	14.95 ± 2.09	4.95 ± 0.79
Sapwood	1.15 ± 0.06	24.91 ± 0.92	46.19 ± 2.23	16.17 ± 0.86	5.29 ± 0.73

The values represent the mean ± SD.

Generally, from the pith outward, the fiber length increases rapidly and then tends to be stable after reaching a mature age (Luo *et al.* 2015). In addition, the values of the fiber width, length-to-width ratio, lumen diameter, and double cell wall thickness of the fiber in the heartwood region were less than these same values in the sapwood.

The cell type proportion of *Toona* sp. wood is presented in Table 2. The fiber proportion of heartwood (73.45%) was slightly greater than that of sapwood (72.68%). One possible reason was that the average fiber lumen diameter of the heartwood is smaller than that of the sapwood, as shown in Table 1. There was a higher proportion of vessels in the heartwood than in sapwood. In addition, the ratio of sapwood wood rays and axial parenchyma cells were greater than those of the heartwood.

Table 2. Cell Type Proportion of *Toona* sp. Wood

Zone	Cell Type Proportion (%)			
	Vessel	Ray	Parenchyma	Fiber
Heartwood	12.15 ± 5.00	10.91 ± 2.08	3.56 ± 0.82	73.45 ± 5.00
Sapwood	10.84 ± 5.64	11.45 ± 2.89	3.70 ± 0.85	72.68 ± 5.64

The values represent the mean ± SD.

Effect of Accelerated Aging on *Toona sp*. Wood Properties

Main chemical compositions

The main chemical components of *Toona sp.* wood before and after accelerated aging treatment are presented in Table 3. The ash content and toluene-alcohol extractives of the aged samples were lower than those of the controls. This result indicated that low-molecular weight components, such as tannin, gums, silicates, and starches, in aged samples were purged by hot water during the water and steam treatment cycles, which may contribute to the reduction in the basic density (Huang *et al*. 2014). The toluene-alcohol extractives of heartwood were greater than sapwood, which was also reported by Shupe *et al*. (1997). One possible reason was that the transformation of sapwood to heartwood could result in the death of the parenchyma cells, and the accumulation and/or deposition of some organic compounds (Bergström 2003).

Table 3. Chemical Components of the Wood Samples Before and After Accelerated Aging Treatment

Chemical Components	Heartwood		Sapwood	
	Control	Aged	Control	Aged
Ash (%)	1.47 ± 0.15	1.10 ± 0.06	0.70 ± 0.06	0.63 ± 0.03
Toluene-alcohol extractives (%)	4.32 ± 0.07	3.05 ± 0.08	3.25 ± 0.09	2.03 ± 0.06
Alpha-cellulose (%)	45.66 ± 0.12	43.17 ± 0.2	44.06 ± 0.26	42.20 ± 0.09
Hemicellulose (%)	25.45 ± 0.31	23.34 ± 0.04	24.46 ± 0.36	22.20 ± 0.12
Klason lignin (%)	27.96 ± 0.01	28.67 ± 0.34	24.75 ± 0.39	26.49 ± 0.27
1% NaOH soluble(%)	20.30 ± 0.25	24.97 ± 0.38	18.47 ± 0.8	21.51 ± 0.1

The values represent the mean ± SD.

As shown in Table 3, higher contents of alpha-cellulose and hemicelluloses were observed in the control samples. In terms of hemicellulose, the reduction in aged samples was higher than cellulose (2.0% to 3.26% and 8.29% to 9.24% for cellulose and hemicellulose, respectively), which can be attributed to fact that hemicellulose is less stable to acid hydrolysis than cellulose is (Yildiz *et al*. 2006). As the hydroxyl group content of hemicellulose decreased, wood became less hydrophilic, resulting in a better dimensional stability (Kránitz *et al*. 2016).

The Klason lignin content in aged samples was higher than that of control samples (Table 3). A similar result was also reported by Shafizadeh and Chin (1977). This was possibly due to the degradation of the carbohydrates, leading to a relative increase of lignin content (Huang *et al*. 2014). The 1% NaOH solubility in aged samples was higher than that of the controls. The 1% NaOH solubility of wood contained primarily extraneous components, such as acid-soluble lignin, tannins, lipids, low-molecular weight hemicelluloses, and degraded cellulose (Pettersen 1984). Moreover, the content of Klason lignin and 1% NaOH solubility in the heartwood were higher than that of the sapwood. The variation in the Klason lignin of *Toona* sp. wood was in line with the report by Taylor *et al.* (2002), who reported more lignin in the heartwood than in the sapwood of *Quercus*. A convincing reason was that transforming sapwood into heartwood could initiate a secondary lignification process (Bergström 2003).

FT-IR analysis

Figure 2 shows the FT-IR spectra of wood samples before and after aging. After the aging treatment, the characteristic infrared (IR) absorption bands of hemicellulose and cellulose became weaker, such as 3330 cm^{-1} (O-H), 1420 cm^{-1} (C-H_2 symmetrical bending), 1370 cm^{-1} (C-H deformation), 1322 cm^{-1} (C-H_2 rocking vibration), 1236 cm^{-1} (C-H_2 tensile, more invasive wood-based ring vibrations), 1205 cm^{-1} (O-H bending), 1157 cm^{-1} (C-O-C asymmetric stretching), 1050 cm^{-1}(C-O vibration), and 899 cm^{-1} (C-H deformation) (Gu *et al.* 2012; Li *et al.* 2015; Fabiyi and Ogunleye 2015; Liu *et al.* 2017). The decompositions of hemicellulose and cellulose, in turn, intensified the lignin characteristic IR peaks, such as 1650 cm^{-1} (C=O tensile) and 1510cm^{-1} (C=C skeleton vibration in aromatic rings) (Li 2003; Liu *et al.* 2017). The increase in methyl and/or methylene infrared bands at 2888 cm^{-1} was noteworthy. This result suggested that macromolecules were decomposed in wood during accelerated aging treatment (Chen *et al.* 2014).

Compared with sapwood, the characteristic IR absorption bands of hemicellulose (1730 cm^{-1} (C=O stretching vibration in non-conjugated ketones and free aldehyde present in lignin and stretching vibration on acetyl and carbonyl groups present in hemicelluloses)), cellulose (1420 cm^{-1} (C-H_2 symmetrical bending), 1322 cm^{-1} (C-H_2 rocking vibration), 1157 cm^{-1} (C-O-C asymmetric stretching), and 899 cm^{-1} (C-H deformation)), and lignin (1650 cm^{-1}(C=O tensile), 1510cm^{-1} (C=C skeleton vibration in aromatic rings)) in heartwood was stronger. This observation was likely a result of higher contents of hemicellulose, cellulose, and lignin in heartwood (Table 3).

Crystallinity of cellulose

The X-ray diffraction (XRD) pattern of the heartwood and sapwood of *Toona sp.* wood before and after accelerated aging are shown in Fig. 3. The crystal peaks at 18°, 22.2°, and 35° represent the diffractive peaks of the crystalline surface of cellulose. Compared with heartwood, the peaks at 18° and 22.2° decreased over time in the sapwood, likely due to the larger crystallized area in the heartwood (Ning *et al.* 2017). The peak intensities at 18° and 22.2° decreased after aging. The accelerated aging samples (58.6% to 58.7%) had a lower relative crystallinity than the control samples (59.9% to 60.4%). This finding suggests that the oxidation of -OH groups in the cellulose and the fracture of the cellulose macromolecular chain caused the destruction of crystalline zones and the transition of some crystalline zones into amorphous zones during the aging treatment (Ning *et al.* 2017). Moreover, the aging treatment promoted the cleavage of glycosidic bonds, resulting in the depolymerization of carbohydrates (Bhuiyan *et al.* 2000). The changes in crystallinity caused by an aging treatment may result in corresponding changes in physical-mechanical and chemical properties.

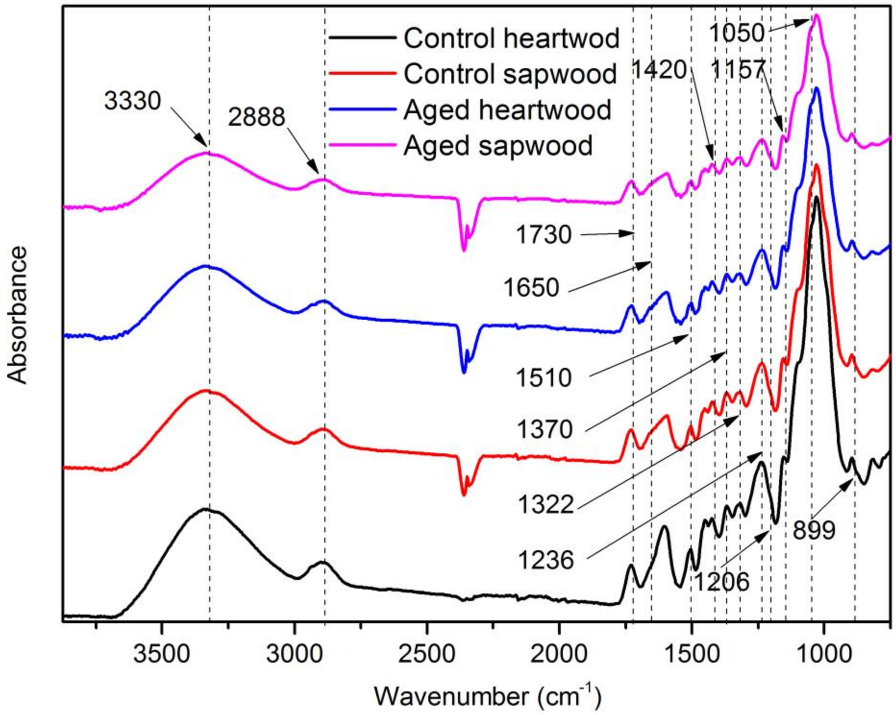

Fig. 2. FT-IR spectra of the wood samples before and after an accelerated aging treatment

Physical and mechanical properties

The physical-mechanical properties of the wood samples are presented in Table 5. The aged samples had a lower basic density and volumetric shrinkage than those of the controls. During the accelerated aging cycles, the reduction of cellulose and hemicellulose content induced by the heating and steaming treatments might contribute to the decreases in basic density and volumetric shrinking (González-Peña *et al.* 2009).

Compared with the sapwood, higher physical-mechanical properties were found in the heartwood, which was probably attributed to the higher density. The physical-mechanical properties were affected by anatomical characteristics, *e.g.*, fiber proportion and cell wall thickness (Pirralho *et al.* 2014). Thus, the observed higher density at the heartwood was mostly a result of the higher fibrous tissue percentage (Table 2). In addition, the volumetric shrinking for heartwood (10.1% to 12.21%) was less than sapwood (12.52% to 13%). This result suggests that heartwood has a higher dimensional stability. The pits between cells usually became tightly sealed during the heartwood formation from sapwood, which greatly reduced wood permeability (Taylor *et al.* 2002). Moreover, the hydrophobic polyphenols compounds in heartwood in the cell wall enhanced the dimensional stability (Taylor *et al.* 2002; Bertaud and Holmbom 2004).

The mechanical properties of the aged samples were lower than those of the control. The results showed that CS, MOE, MOR, and hardness of aged samples decreased by 7.3% to 10.3%, 3.8% to 7.3%, 11.8% to 12.2%, and 8.67% to 10.88%, respectively. The primary reason for the strength loss was the degradation of cellulose and hemicelluloses (Hillis 1984). In addition, the decreased crystallinity of cellulose influenced the mechanical properties as well (Bhuiyan *et al.* 2000). Based on these results, it is easy to understand that the reduction in mechanical properties with accelerated aging had a positive relationship in regards to the loss of carbohydrate components.

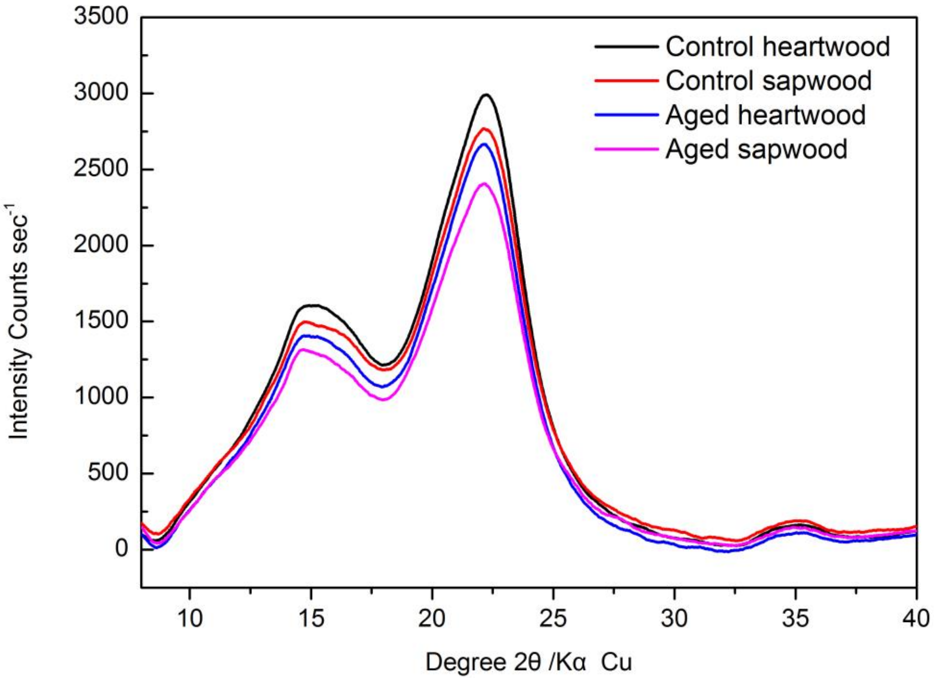

Fig. 3. XRD spectra of the wood samples before and after the accelerated aging treatment

In comparison to the sapwood, the heartwood had higher mechanical properties. These findings were similar with the conclusions reached by Sun *et al.* (2012). The higher mechanical properties of heartwood may be attributed to the higher content of main chemical components in the cell wall as shown in Table 3.

Table 4. Relative Crystallinity of the Wood Samples Cellulose Before and After Accelerated Aging Treatment

Item	Heartwood		Sapwood	
	Control	Aged	Control	Aged
Relative crystallinity (%)	59.89	58.67	60.41	58.59

Table 5. Physical and Mechanical Properties of the Wood Samples Before and After Aging Treatment

Physical and Mechanical Properties		Heartwood		Sapwood	
		Control	Aged	Control	Aged
D (g•cm^{-3})		0.476±0.01	0.457±0.02	0.472±0.02	0.447±0.03
S (%)		12.21±0.29	10.10±0.23	13.00±0.34	12.52±0.15
CS (MPa)		31.34±3.49	29.05±4.37	29.57±2.73	26.53±2.79
MOR (MPa)		64.96±6.99	62.51±7.88	57.94±8.58	52.91±2.31
MOE (GPa)		8.34±0.14	7.32±0.10	6.55±0.38	5.78±0.19
Hardness (KN)	Cross section	4.32±0.58	3.85±0.25	3.92±0.54	3.58±0.19

The values represent the mean ± SD.

CONCLUSIONS

1. The physical-mechanical properties of heartwood were higher than those of sapwood, which corresponded to the variation in anatomical characteristics and chemical components.
2. The accelerated aging treatment tended to weaken the physical-mechanical properties of the wood samples.
3. The carbohydrates (cellulose and hemicelluloses) were depolymerized during the aging treatments, which resulted in a reduction in physical-mechanical properties.
4. In addition, the reduction of physical-mechanical properties was also a result of the decreasing cellulose crystallinity.

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