Effect of Hot-water Extraction on the Hygroscopicity, Shrinkage, and Swelling of *Paulownia tomentosa* Wood

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Liquid water and water vapor absorption on each separate surface and all surfaces at once, as well as the oven-dry shrinkage before and after the hot-water extraction of the wood, were examined using Korean Standards. The extractives content was examined using American Society for Testing and Materials (ASTM) standards. The highest water absorption and moisture adsorption were found in the transverse sections of both unextracted and extracted wood samples. There was no change in the equilibrium moisture content (EMC) in the unextracted *vs.* extracted wood samples at a relative humidity (RH) of 75%. However, the EMC in the extracted sample was higher than that in the unextracted sample at a RH of 90%. Swelling per 1% moisture content remained unchanged in the radial direction before and after extraction, while it increased in the tangential direction. The linear and volumetric shrinkage, as well as the coefficient of anisotropy, considerably increased after extraction. There was no difference in the extractive content between sapwood and heartwood. In conclusion, it was confirmed that the extractives in wood considerably affect the hygroscopicity and shrinkage of *Paulownia* wood.

Keywords: Paulownia tomentosa; Extractives; Hygroscopicity; Water-absorption; Moisture-adsorption; Shrinkage; Swelling

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INTRODUCTION

Korea imports approximately 83.7% of its domestic wood, due to the high global demand of the wood. Moreover, the country faces difficulties associated with timber supply and demand, due to the policies of the timber-exporting countries and their exchange rates. Therefore, the Korean government has established a policy aimed at achieving a timber self-sufficiency of 50% by 2050, and it is making significant efforts for afforestation in both domestic and foreign countries (Korea Forest Service 2008). Afforestation in Korea requires more than 30 years to result in economically valuable wood resources. To overcome this problem, cultivation of fast-growing domestic trees has been deemed necessary.

The *Paulownia tomentosa* tree is one of the fast-growing trees in Korea. It grows up to approximately 20 m in height and approximately 0.8 m in diameter. Moreover, it can be used as timber 15 to 20 years after planting because it grows three times faster than other species.

To obtain valuable information on the complete utilization of *Paulownia* trees, the anatomical, physical, and mechanical properties of stems, branches, and roots of the trees were investigated (Qi *et al*. 2014, 2016a,b). The effects of heat treatment on wood properties (Hidayat *et al*. 2017a,b; Kim *et al*. 2018b; Kang *et al*. 2019; Kim and Kim 2019) and the bioenergy characteristics of *Paulownia* wood (Qi *et al*. 2016a,c) were also studied.

Paulownia wood has been used for various purposes by Koreans. Examples of its applications include the manufacturing of musical instruments, furniture and appliance materials, and household goods. The wood has a low specific gravity with a thin cell wall and high sound-exchange ability; these properties are suitable for musical instruments (Kang *et al*. 2019). It also has high dimensional stability, excellent machinability, and favorable properties for particleboards or pulp (Chong and Park 2008; San *et al*. 2016). In addition, its characteristic rot resistance, dimensional stability, and high ignition point contribute to the popularity of this timber in the world market (Akyildiz and Kol 2010).

Paulownia tomentosa wood contains high amounts of extractives compared to other common hardwoods that are grown in Korea (Chong and Park 2008). The major extractives of *Paulownia tomentosa* wood are d-sesamin, syringin, paulownin, and eleostearic acid (He *et al*. 2016). Generally, extractives in wood are a major contributor to many properties of wood despite their lower amounts and volumes.

There have been many studies on the effect of extractives to hygroscopicity and shrinkage in different wood species, such as the southern pine woods from the United States (Choong 1969), Argentine wood species (Turc and Cutter 1984), Chile wood species (Popper *et al.* 2007), tropical wood species (Choong and Achmadi 1991; Jankowska *et al*. 2017), *Sequoia sempervirens* (Demaree and Erickson 1975), *Robinia pseudoacacia* (Adamopoulos 2002; Adamopoulos and Voulgaridis 2003, 2012), *Prunus serotine*, *Querqus rubra*, *Pinus resinosa* (Nzokou and Kamdem 2004), and *Pinus sylvestris* (Vahtikari *et al*. 2017).

Thus far, little information is available on the hygroscopicity and dimensional stability of extractives of *P. tomentosa* wood. Therefore, in this study, the effects of extractive removal on the hygroscopicity, shrinkage, and swelling of wood were studied to obtain valuable information on its further effective utilization.

EXPERIMENTAL

Materials

Three trees of *P. tomentosa* (Thunb.) Steud. that were 15 to 20 years old were harvested from the research forest at Kangwon National University, Chuncheon, Korea (N 37° 77', E 127° 81'). Table 1 shows the characteristics of the sample trees.

Table 1. Basic Information of Sample Trees

Species	Age (Years)	Height (m)	D.B.H. (cm)	Density (g/cm^3)	Average Growth Ring Width (mm)	Location
Paulownia tomentosa	15	13	33.0	0.32	7.85	Chuncheon, Korea (N 37° 77', E 127° 81')
	18	15	42.5	0.34	9.10	
	20	17	43.0	0.33	10.10	

Notes: D.B.H. = Diameter at breast height; Specimen of density was calculated with whole part.

Methods

Table 2 shows the experimental information for each property of unextracted and extracted *Paulownia* wood. All wood samples were prepared from the 8th to 15th growth ring of the heartwood at breast height in all trees. To remove the extractives, the samples were boiled in distilled water at 100 °C for 75 h, maintained for 2 days at room condition at 24±2 °C, and then oven-dried at 103±2 °C. After hot-water extraction, the samples were kept in a desiccator for cooling, and then the weight loss was observed.

Table 2. Sample Preparation for Each Measurement

State	Measurement (Standard)	Number of Sample	Sample Dimension (mm)	Weight Loss After Extraction (%)
Unextracted and Extracted wood samples	Water absorption (KS F 2204 (1999))	Transverse 20 (20) Radial 20 (20) Tangential 20 (20)	30 × 30 × 100 (L)	5.1 (±0.4)
	Hygroscopicity (KS F 2205 (2004))	Transverse 20 (20) Radial 20 (20) Tangential 20 (20)	30 × 30 × 60 (L)	9.8 (±0.7)
		Entire surface 20 (20)	30 × 30 × 5 (L)	8.3 (±0.03)
	Shrinkage (KS F 2203 (2004))	20 (20)	20 × 20 × 30 (L)	8.5 (±0.1)

Notes: Numbers in parentheses are extracted samples; L = Longitudinal direction

Water absorption

The water absorption measurement was carried out according to the KS F 2204 (1999) standard. All surfaces of the oven dried samples were coated using a mixture of paraffin and Vaseline except for the measuring surface, and then the samples were soaked in distilled water at 25 ± 1 °C for 24 h. The water absorption was calculated using Eq. 1,

$$W_{ab} = \frac{W_2 - W_1}{A}\ (\mathrm{g/cm^2}) \tag{1}$$

where W_{ab} (g/cm^2) is water absorption, W_1 (g) is the weight after coating the oven-dried samples, W_2 (g) is the weight after soaking the coated samples, and A (cm^2) is the absorption area.

Moisture adsorption on each surface

The moisture adsorption measurement on transverse, radial and tangential surfaces was carried out according to the KS F 2205 (2004) standard. The wood samples with constant weight at a relative humidity (RH) of 75 ± 2% and a temperature of 40 ± 1 °C in a thermohygrostat (TH3-PE, JEIO TECH, Daejeon, Korea) were coated with a mixture of Vaseline and paraffin, except for the measuring surface. The wood samples were then placed under a RH of 90 ± 2% and a temperature of 40 ± 1 °C for 24 h in the chamber. The moisture adsorption was calculated using Eq. 2,

$$M_{ad} = \frac{W_{24\mathrm{h}} - W_{0\mathrm{h}}}{A}\ (\mathrm{g/cm^2}) \tag{2}$$

where M_{ad} is the moisture adsorption (g/cm^2), W_{0h} (g) is the weight stabilized at a RH of 75% and 40 °C, W_{24h} (g) is the weight without mixture of paraffin and Vaseline after placing the samples in a desiccator at a RH of 90% and 40 °C for 24 h, and A (cm²) is the adsorption area.

Moisture adsorption on the entire surface

The moisture adsorption measurement on the entire surface was carried out according to the KS F 2205 (2004) standard. The constant weight of the wood samples at a RH of 75 ± 2% at 40 ± 1 °C and at a RH of 90 ± 2% at 40 ± 1 °C was obtained in the thermohygrostat. The moisture adsorption (EMC, equilibrium moisture content) and swelling per 1% moisture content were calculated using Eq. 3,

$$\text{EMC at RH 75\%} = \frac{W_{(0h)} - W}{W} \times 100\ (\%)$$

$$\text{EMC at RH 90\%} = \frac{W_{(\infty)} - W}{W} \times 100\ (\%)$$

$$\text{Swelling per 1\% moisture content} = \frac{L_{(t,r)} - l_{(t,r)}}{l_{(t,r)}} \times \frac{W}{W_{(\infty)} - W_{(0h)}}\ (\%) \qquad (3)$$

where W (g) is the oven-dried weight, W_{0h} (g) and l (mm) are the weight and length, respectively, stabilized at a RH of 75% and at 40 °C, W_{∞} (g) and L (mm) are the weight and length, respectively, stabilized at a RH of 90% and at 40 °C.

Shrinkage

The shrinkage was analyzed according to the KS F 2203 (2004) standard. The shrinkage in the oven-dry condition (S_o) was calculated using Eq. 4. The volumetric shrinkage is the sum of longitudinal, radial, and tangential shrinkages.

$$S_o = \frac{l_g - l_o}{l_o} \times 100\ (\%) \qquad (4)$$

where l_g (mm) and l_o (mm) are the lengths in the green (over 100 % moisture content) and oven-dry conditions, respectively.

Extractive content

The extractive contents of the wood in cold-water and hot-water extraction were analyzed according to ASTM D1110 (2013) standard. The extraction process was repeated thrice for heartwood and sapwood. For cold-water extraction, 3 g of oven-dried wood powder was stirred in 300 mL of distilled water and kept in a 400 mL beaker at 23 ± 2 ℃ for 72 h. For hot-water extraction, 3 g of the oven-dried wood powder was stirred and boiled for 3 h in 100 mL of distilled water and kept in a 200 mL round-bottom flask that was placed on a connecting reflux condenser. The treated wood powder was filtered and rinsed with distilled water using a glass filter (1G3), dried at 103 ± 2 ℃, cooled in a desiccator, and then weighed. The extractive content was calculated using Eq. 5,

$$\text{Extractive content} = \frac{W_1 - W_2}{W_1} \times 100\ (\%) \qquad (5)$$

where W_1 (g) and W_2 (g) are the weights of oven-dried wood powder and extracted wood powder, respectively.

Statistical analysis

The statistical differences in the water absorption, moisture adsorption, and shrinkage between unextracted and extracted samples were analyzed using one-way analysis of variance (SPSS ver. 21, IBM Corp., New York, USA).

RESULTS AND DISCUSSION

Water Absorption

Table 3 shows water absorption on each surface of the unextracted and extracted *Paulownia* wood samples. The extracted samples exhibited higher water absorption on all the surfaces than the unextracted samples. Although the amount of water absorption was the highest on the transverse surface after extraction, the increase was 66%, 88%, and 113% on the transverse, radial, and tangential surfaces, respectively. In the extracted sample, the water absorption on the tangential surface was slightly higher than that on the radial surface. The unextracted wood had smaller water absorption in all surface than extracted wood. There were significant differences in water absorption of the three surfaces between unextracted and extracted wood.

Chong and Park (2008) reported the water absorption results of *Paulownia* wood that were similar to the results of this study. That is, they reported a water absorption of 0.191 g/cm² in the transverse surface, 0.046 g/cm² in the radial surface, and 0.058 g/cm² in the tangential surface. Nzoko and Kamdem (2004) suggested that increased water absorption due to extractive content could lead to increased dimensional instability and eventually lead to more cracks and checks in the extracted wood.

Table 3. Water Absorption on the Three Surfaces of *Paulownia* Wood

Water Absorption (g/cm^2)			
Sample	Transverse	Radial	Tangential
Unextracted	0.23 (0.03)^a	0.08 (0.01)^a	0.08 (0.01)^a
Extracted	0.35 (0.03)^b	0.15 (0.01)^b	0.17 (0.02)^b

Notes: The numbers in parentheses are standard deviations. Items having the same letters beside the mean value are insignificant at the 5% significance level between the unextracted and extracted samples.

Moisture Adsorption

Moisture adsorption on each surface

The moisture adsorption on the three surfaces of *Paulownia* wood is shown in Table 4. The moisture adsorption of the unextracted samples was the highest on the transverse surface with a value of 0.022 g/cm². The radial and tangential surfaces exhibited the same amount of moisture adsorption with a value of 0.009 g/cm². After extraction, the moisture adsorption on the three surfaces increased 36% on the transverse surface and 89% on the radial and tangential surfaces. The moisture adsorption of unextracted wood in each surface was significantly smaller compare to that of extracted wood. The total moisture adsorption of extracted wood increased 1.6 times relative to the unextracted wood sample.

Table 4. Moisture Adsorption on the Three Surfaces of *Paulownia* Wood

Moisture Adsorption (g/cm^2)					
Sample	Transverse	Radial	Tangential	Total	E/UE
Unextracted	0.022 (0.002)^a	0.009 (0.001)^a	0.009 (0.001)^a	0.040	1.6
Extracted	0.030 (0.002)^b	0.017 (0.001)^b	0.017 (0.001)^b	0.064	
Notes: The numbers in parentheses are standard deviations; E = extracted, UE = unextracted; The same letters beside the mean value are insignificant at the 5% significance level between the unextracted and extracted samples.					

Moisture adsorption on the entire surface

Table 5 shows the moisture adsorption on the entire surface of *Paulownia* wood. The EMC in unextracted wood at a RH of 75% and a RH of 90% was 9.6% and 13.9%, respectively. During the EMC change from a RH of 75% to a RH of 90%, the dimensional change per 1% moisture content in unextracted wood was 0.05% on the radial surface and 0.09% on the tangential surface. Moreover, the anisotropy coefficient reflecting tangential *vs.* radial shrinkage (T/R) was 2.06. After extraction, the EMC on the radial and tangential surfaces at a RH of 75% and a RH of 90% was 9.7% and 15.2%, respectively. There was no EMC change in both extracted and unextracted wood at a RH of 75%. However, the extracted sample at a RH of 90% exhibited higher EMC than the unextracted counterpart. There was a significant difference in EMC at RH of 90% between unextracted and extracted wood. Swelling per 1% moisture content showed no significant difference in the radial direction of unextracted and extracted wood. However, it significantly increased by approximately 2 times in the tangential direction after extraction. In addition, The EMC in at RH 75% of unextracted wood showed no significant difference to that of extracted wood, while there was significant difference between unextracted and extracted wood in EMC at RH 90%. Moreover, the anisotropic coefficient increased considerably after extraction. As a result, it was confirmed that the extractives noticeably affect the moisture adsorption of *Paulownia* wood.

The EMC of 25 hardwood species ranged from 9.7% to 12.7% at a RH of 75% and from 13.6% to 17.9% at a RH of 90% (Kim *et al.* 2018a). Turc and Cutter (1984) also found that the moisture adsorption of six Argentine wood species was in the range 15.5% to 19.5% at a RH of 90% and 20 °C. In agreement with these previous reports, the *Paulownia* wood in this study exhibited slightly lower EMC than other species.

The effect of moisture on wood properties and behaviors is closely related to the chemical composition and biological structure of the wood (Treschsel and Bomberg 2009). The moisture adsorption of unextracted and extracted wood appeared to be remarkably different at a high RH. At a high RH, the extracted samples adsorbed more moisture than the unextracted samples (Choong and Achmadi 1991; Nzoko and Kamdem 2004). The enhanced response to RH changes in extracted wood is probably due to the reduced bulking of the cell wall because of the removal of extractives (Song *et al*. 2014; Vahtikari *et al*. 2017). Moreover, it has been suggested that extractives form a coating layer in the cell wall and pits, thus interfering with the permeability of the wood (Krahmer *et al*. 1970; Hillis 1971).

Table 5. EMC and Swelling Per 1% Moisture Content in *Paulownia* Wood

Sample	EMC (%) at 40 °C		Swelling Per 1% Moisture Content (%)		
	RH 75%	RH 90%	Radial	Tangential	AC(T/R)*
Unextracted	9.6 (0.3)[a]	13.9 (0.6)[a]	0.050 (0.01)[a]	0.103 (0.03)[a]	2.06
Extracted	9.7 (0.4)[a]	15.2 (0.2)[b]	0.051 (0.02)[a]	0.202 (0.07)[b]	3.96

Notes: The numbers in parentheses are standard deviations; The same letters beside the mean values indicate that differences are insignificant at the 5% significance level between the unextracted and extracted samples. * AC(T/R) = anisotropy coefficient

Shrinkage

Table 6 shows the shrinkages of unextracted and extracted *Paulownia* wood. The shrinkage of unextracted wood was 3.91% in the tangential direction, 1.67% in the radial direction, and 0.23% in the longitudinal direction. The ratio of shrinkage on different directions of unextracted samples was 10 (tangential, T): 4.3 (radial, R): 0.6 (longitudinal, L), and the anisotropy coefficient was 2.34. The shrinkage of extracted wood increased 26% in the longitudinal direction, 29% in the radial direction, 62% in the tangential directions, and 50% in volume compare to unextracted wood. The radial, tangential and volumetric shrinkage of unextracted wood were significantly different to those of extracted wood, while there was no significant difference between unextracted and extracted sample in longitudinal shrinkage. The anisotropy coefficient also increased considerably. There were considerable differences in the shrinkage and the anisotropy coefficient between the unextracted and extracted woods.

Extractives located in the cell lumen reduce porosity and increase specific gravity. During extraction, extractives could be removed with free water and could affect the shrinkage of wood (Demaree and Erickson 1975). Tsoumis (1991) explained that a large extractive content reduces shrinkage and swelling, and the removal of extractives increases shrinkage and swelling. According to Mantanis *et al*. (1994) and Adamopoulos and Voulgaridis (2012), extractives deposited in the cell wall structure have a definite influence on the swelling and shrinkage of the cell wall, and therefore, would affect the swelling and shrinkage of wood as evidenced by the changes in external dimensions. Choong and Achmadi (1991) also reported that the removal of extractives facilitates the availability of additional moisture sorption sites, and therefore, it causes an increase in shrinkage.

Table 6. Shrinkage from Green to Oven-dry of *Paulownia* Wood

Sample	Shrinkage (%)				
	Longitudinal	Radial	Tangential	Volume	AC(T/R)*
Unextracted	0.23 (0.40)[a]	1.67 (0.30)[a]	3.91 (0.29)[a]	5.79 (0.69)[a]	2.34
Extracted	0.29 (0.13)[a]	2.15 (0.21)[b]	6.35 (0.51)[b]	8.67 (0.70)[b]	2.95

Notes: Numbers in parentheses are standard deviations; The same letters beside the mean value are insignificant at the 5% significance level between the unextracted and extracted samples. * AC(T/R) = anisotropy coefficient

Extractives Content

Table 7 shows the extractive content of heartwood and sapwood of *Paulownia* wood. During cold-water extraction, the extractive content was 9.11% in heartwood and

8.97% in sapwood. In hot-water extraction, it was 10.76% in heartwood and 10.14% in sapwood. It is evident from these results that there was no significant difference in the extractive contents between heartwood and sapwood. In addition, the extractive content by hot-water extraction was slightly higher than that by cold-water extraction.

Tannins, gums, sugars, and coloring matter are extracted by cold-water extraction, while hot-water extraction removes starch with the extractives by cold-water extraction (ASTM D1110-84 2013). In this study, the extractive content of *Paulownia* wood was higher than reported in previous studies (Olson and Carpenter 1985; Chong and Park 2008). Moreover, *Paulownia* wood exhibited higher extractive content compared to other hardwood species in Korea (Chong and Park 2008). Olson and Carpenter (1985) reported that hot-water extractives of both sapwood and heartwood were similar in young *Paulownia* wood, and more than twice the total extractives were found in the sapwood.

Table 7. Extractives Content in Heartwood and Sapwood of *Paulownia* Wood

Extractive Content (%)			
Cold-water	HW	9.11 (1.12)	9.04 (0.92)
	SW	8.97 (0.67)	
Hot-water	HW	10.76 (1.32)	10.45 (1.16)
	SW	10.14 (0.87)	

Notes: Means represent averages of three replicates and numbers in parentheses are standard deviations; HW = Heartwood, SW = Sapwood;

CONCLUSIONS

1. The extracted wood samples exhibited higher liquid water absorption and water vapor adsorption on each surface than the unextracted wood samples. The water absorption and water vapor adsorption after extraction increased considerably on the radial and tangential surfaces.
2. The equilibrium moisture content (EMC) in both extracted and unextracted woods was similar at a RH of 75%, while the extracted wood exhibited higher EMC than the unextracted wood at a RH of 90%. Swelling per 1% moisture content did not change in the radial direction before and after extraction but increased approximately twice in the tangential direction. Moreover, the anisotropy coefficient (T/R) increased considerably after extraction.
3. The linear and volumetric shrinkage and anisotropy coefficient increased considerably after extraction.
4. Extractive contents in the heartwood and sapwood of *Paulownia* wood were the same, and they were approximately 10% when hot-water extraction was employed.

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