Changes in Physical Properties of Sugi, Hinoki, and Korean Pine Wood after Fire-retardant Treatment

Mingyu Wen,[a,b] Yaoxing Sun,[a,]* Junyou Shi,[a,]* and Heejun Park [c]

The effect of a fire-retardant treatment on some physical properties, including dimensional stability, hygroscopicity, and surface color variation of Sugi (*Cryptomeria japonica*), Korean Pine (*Pinus koraiensis*), and Hinoki (*Chamaecyparis obtusa*) were investigated in this study. These softwoods were subjected to vacuum-pressure and impregnated with a developed fire-retardant chemical. The results showed that the radial and tangential swelling of 1% moisture content (MC) increment were lower for all three wood species compared with the control specimen after the fire-retardant treatment, despite higher equilibrium moisture content (EMC) at 75% relative humidity (RH) and 90% RH at 40 °C. Meanwhile, the bulk coefficient after water immersion decreased for all specimens after the treatment, which indicated higher dimensional stability. However, the fire-retardant treatment proved a shift in surface color to darkness.

Keywords: Fire-retardant wood; Dimensional stability; Hygroscopic behavior; Color variation

*Contact information: a: Wood Material Science and Engineering Key Laboratory, Beihua University, Jilin 132013 China; b: Key Laboratory of Biobased Polymer Materials, Shandong Provincial Education Department, School of Polymer Science and Engineering, Qingdao University of Science and Technology, Qingdao266042 China; c: Department of Housing Environmental Design, and Research Institute of Human Ecology, College of Human Ecology, Chonbuk National University, Jeonju 561-756 Korea; *Corresponding author: jlwenmingyu@163.com*

INTRODUCTION

Wood can provide good service across many industries under proper use conditions. However, in unfavorable conditions, it may easily be damaged and destroyed by fire. Therefore, the treatment of wood with a fire retardant is a crucial precautionary measure. The proper application of fire-retardant chemicals can protect wood from fire, thus prolonging the service life of wood. Many studies have investigated the effectiveness of various fire-retardant treatments on wood (Dubey *et al.* 2012).

The green nitrogen-phosphorus (N-P) fire retardant shows many advantages, such as environmentally friendly character, non-toxicity, low cost, and excellent fire-retardant performance. Meanwhile, nitrogen phosphorous compounds that could redirect decomposition reactions in favor of reactions yielding carbon over carbon monoxide have received more attention than ever, both in an academic and industry field (Huo *et al.* 2016; Chu *et al*. 2017a,b). Most research has focused on the fire-retardant properties such as thermal degradation and combustibility (Li *et al*. 2000; Gao *et al*. 2005; Lowden and Hull 2013; Jin and Chung 2016).

However, research on N-P fire retardants has been limited due to the high hygroscopicity and poor smoke-suppression effects. It has been recognized that the non-fixed characteristics of the flame retardant and simply depositing it in the wood cell lumen leads to their leaching out with liquid water or moisture contact (Jiang *et al*. 2015). Consequently, the development of a flame retardant that can fulfill the requirement of flame retardancy and have the least adverse influence on the physical properties of wood is urgently needed. Thus, considerable research has been conducted on the influence of various treatments on the physical and hygroscopic properties of wood. (Cai *et al*. 2007; Kamperidou *et al*. 2013). For instance, Pan *et al.* (2014) found that the combination of N-P fire retardant and 10% $Mg(OH)_2$/$Al(OH)_3$ was more effective for smoke suppression and char residual yield increased. By impregnating nano-SiO_2 into fire-retardant-treated poplar wood, the hygroscopicity was greatly decreased. Chu *et al.* (2017a,b) investigated the effects of the combined N-P treatment and heat treatment (HT). They concluded that N-P pre-treatment intensified the effectiveness of HT, and dimensional stability of HT wood was improved by N-P pre-treatment. However, expectedly, the reactions between N-P and wood components lead to a color change.

In our preliminary studies, the variation in impregnation between and within wood species was investigated. And the regression function of chemical uptake as a function of time for different species, positions, and pressure by statistic method was established, which provided theoretical and technical basis for preparing fire retardant wood with proper process. The effects of the fire-retardant treatments on mechanical properties and fire retardant performances were determined. We found that the modulus of rupture (MOR) and static modulus of elasticity (MOE) of three wood species decreased; conversely, the dynamic modulus of elasticity (DMOE) increased as a result of fire retardant treatment. In addition, the physical pre-treatment method for wood fire retardant impregnation was developed and the fire-retardant performance was assessed, in an attempt to apply it to reconstruct traditional wooden structure poles and beams. After comprehensive evaluations of chemical uptake, the boring and combination methods with a boring diameter less than 12 mm were recommended (Wen *et al*. 2014; Park *et al*. 2015; Kang *et al*. 2017; Park *et al*. 2017).

Following the previous research, this paper focuses on evaluating the effect of the N-P fire retardant treatment on some physical properties of wood, including dimensional stability and color variation. Three softwoods typical widely used in wooden buildings and decoration in Korea, including Sugi (*Cryptomeria japonica*), Korean pine (*Pinus koraiensis*), and Hinoki (*Chamaecyparis obtusa*) were selected. In order to fulfill the utilization requirement of high quality of these three wood species, it is essential to offer specific modification approaches for wide application. In addition, generally, most N-P fire retardants have hydroscopic character, resulting in leaching out and adverse effect on dimensional stability. In our research the combination of flame retardant and guanylurea phosphate (GUP) and some additives are expected to offer synergistic effects for improvement of wood properties both for flame retardancy and dimensional stability.

EXPERIMENTAL

Materials

Wood specimen preparation

Three softwood species, including Sugi (*Cryptomeria japonica*), Korean pine (*Pinus koraiensis*), and Hinoki (*Chamaecyparis obtusa*) were purchased from Happy Home Wood Tech. Co., Ltd. (Mokpo City, Korea), and were selected for this study. Prior to treatment, the squares were kiln-dried to 8% moisture content (MC) and then labeled and weighed. For the measurement of dimensional stability, the specimens were then cut into 30 mm (T) × 30 mm (R) × 5 mm (L) samples, which totals 20 specimens for each sample.

Fire-retardant Resin Impregnation

A new water-soluble composite nitrogen-phosphorus fire retardant chemical was used in this study. Its main components were ammonium phosphate polymer (APP), guanylurea phosphate (GUP), and a minor amount of additives. The resin content and specific gravity of this chemical were 25 wt.% and 1.13, respectively. Wood samples were randomly placed in a vertical cylindrical vessel under a pressure of -0.098 MPa for 5 min to remove the air contained within the wood prior to impregnation. When the vacuum was released, the fire-retardant chemicals refluxed into the vessel, and the pressure was raised to 10 kgf/cm^2 for 20 min. Then, the pressure was released and the specimens were removed and weighed. After impregnation, the specimens were air-dried for two weeks and then cured at 60 °C for 72 h to a MC of 12%.

Methods

Dimensional stability - Equilibrium moisture content (EMC) at 75% relative humidity (RH) and 90% RH

The specimens were stored at a temperature of 40 °C and a RH of 75% and 90%, until the samples reached the equilibrium moisture content (EMC), which was considered to be reached when the mass change of the samples within 24 h was less than 1% of the contained water. Moisture uptake was related to the volume of the dry specimens.

Radial and tangential 1% moisture content (MC) swelling ratio

When the MC increased 1%, the average swelling on the tangential and radial directions was calculated according to Eqs. 1 and 2,

$$\text{Tangential 1\% MC swelling (\%)} = \left[\frac{L_{T90}-L_{T75}}{L_{T75}}\right] \times \left[\frac{W_0}{W_{90}-W_{75}}\right] \times 100 \quad (1)$$

$$\text{Radial 1\% MC swelling (\%)} = \left[\frac{L_{R90}-L_{R75}}{L_{R75}}\right] \times \left[\frac{W_0}{W_{90}-W_{75}}\right] \times 100 \quad (2)$$

where L_{T90} and L_{T75} are the length of specimen (mm) in the tangential direction with the EMC at 40 °C and 75% RH, L_{R90} and L_{R75} are the length of specimen (mm) on the radial direction with the EMC at 40 °C and 90% RH, W_0 is the oven-dried weight of the specimen (mm), W_{90} and W_{75} are the weight of the specimen with EMC at 40 °C and 75% RH and 90% RH, respectively.

Hygroscopic behavior

The bulking coefficient (B) can be defined as follows,

$$B(\%) = \frac{a_1 - a_0}{a_0} \times 100 \quad (3)$$

where a_0 and a_1 are the oven-dried cross-sectional area (mm) before and after the treatment, respectively.

The specimens were conditioned at 20 °C and 90% RH for about 20 days. The anti-swelling efficiency (ASE_m) in the conditioned state was calculated from the coefficient of cross-sectional swelling at 20 °C and 90% RH on the basis of the oven-dried state. Especially for the treated specimens, the coefficient of swelling (S_m^t) was calculated from the oven-dried cross-sectional area after once saturated in water (a_3) and the conditioned (20 °C and 90% RH) cross-sectional area (a_4) as follows:

$$\mathrm{ASE_m}(\%) = \frac{S_m^u - S_m^t}{S_m^u} \times 100 \quad (4)$$

$$S_m^t(\%) = \frac{a_4 - a_3}{a_3} \times 100 \quad (5)$$

Color changes

A colorimeter (type CR-400/410, KONICA MINOLTA, Tokyo, Japan) with D65 light source was used to quantify color change at the surface of the specimen before and after the fire-retardant treatment. Color changes determination was conducted on tangential faces. The CIE $L^*a^*b^*$system which works according to the CIE standard (ASTM D2244-07 (2007)) provides a standard scale for the comparison of color values. The L^* coordinate describes the lightness and ranges up to 100, which represents a perfect reflecting diffuser, and 0, which represents black; a^* and b^* describe the chromatic coordinates on the red-green and blue-yellow axes, respectively, without specific numerical limits. The three color coordinates, L^*, a^*, and b^*, were recorded before and after each fire-retardant treatment and the values were used to calculate the total color difference (ΔE), the metric chroma (C^*), and the saturation (ΔC). The color change E^*_{ab} was calculated according to the following equation:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + [(\Delta a^*)^2 + [(\Delta b^*)^2]^{1/2} \quad (6)$$

$$C^* = [(a^*)^2 + (b^*)^2]^{1/2} \quad (7)$$

RESULTS AND DISCUSSION

Dimensional Properties of Fire-retardant Wood

EMC and swelling

The dimensional properties of the three fire retardant-treated woods were evaluated by EMC at 75% RH and 90% RH and swelling studies. The results are shown in Tables 1 and 2. After the fire-retardant treatment, the EMC of Sugi, Hinoki, and Korean pine increased compared to the controls. For Sugi, Hinoki, and Korean pine, at 75% RH, the EMC of specimens with treatment increased 33%, 18%, and 101%, respectively, and at 90% RH, the EMC of specimens with treatment increased 55%, 74%, and 182%, respectively. This indicated that the hygroscopicity of wood increased after fire-retardant treatment. When hygroscopicity increased, the swelling of the wood was supposed to increase. However, the results of the specimens after treatment were approximately 10% to 25% lower than that of the control specimens by calculation of the radial and tangential swelling of 1% MC increments. The changes in the dimensions in the tangential and radial direction in solid wood are a great problem for wood. The property with a higher hygroscopicity and lower swelling is desired in the view of wood products application. The results implied that in higher humidity, wood is able to adjust by absorbing humidity with lower swelling.

The dimensional stability of fire-retardant wood is also dependent upon the hygroscopicity of the chemical used. It was considered that the high hygroscopicity of the samples after treatment was attributable to the high hygroscopicity of the N-P fire retardant, which contains APP, GUP, and phosphoric acid.

Table 1. Dimensional Stability Evaluation of Sugi, Hinoki, and Korean Pine

	Specimen	Number	EMC 40 °C, 75% RH (%)		EMC 40 °C, 90% RH (%)	
			Mean	Std	Mean	Std
Sugi	Control	20	11.39	0.40	14.91	1.82
	Treatment	20	15.17	0.62	23.11	2.20
Hinoki	Control	20	11.92	0.54	14.31	0.71
	Treatment	20	14.10	0.42	25.04	0.65
Korean pine	Control	20	9.67	0.74	11.84	0.94
	Treatment	20	19.49	0.82	33.35	2.55

Table 2. Radial and Tangential Swelling of 1% MC Increment

Species	Specimen	N	Radial 1% MC swelling (%)		Tangential 1% MC swelling (%)	
			Mean	Std.	Mean	Std.
Sugi	Control	20	38.43	10.76	24.34	4.45
	Treatment	20	3.81	2.39	3.79	1.14
Hinoki	Control	20	21.79	12.80	36.92	6.94
	Treatment	20	2.36	1.09	1.18	1.05
Korean pine	Control	20	47.28	31.25	30.17	16.96
	Treatment	20	1.92	2.18	1.46	1.15

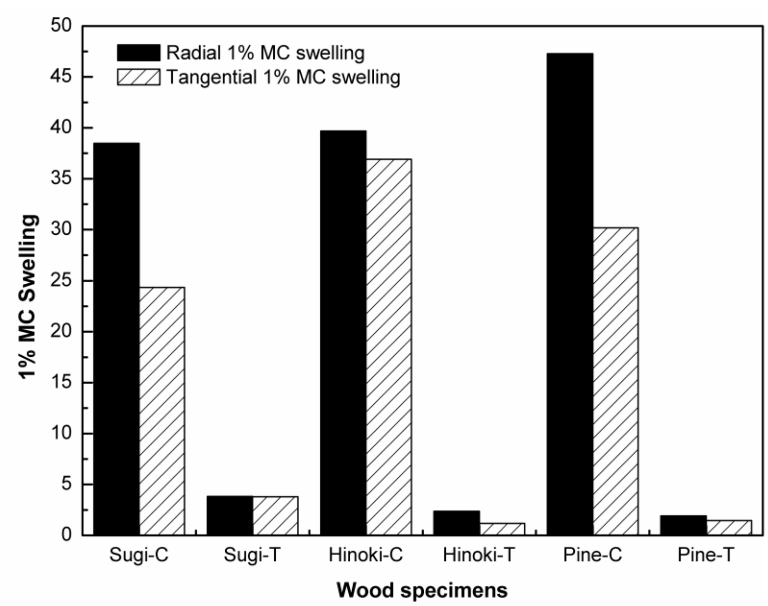

Fig. 1. Radial and tangential swelling with 1% MC increase

The reduced swelling resulted from the reaction of the fire retardant and wood cell wall, which caused the hemicelluloses to change during the drying process. Repellin and Guyonnet (2005), who evaluated the heat-treated wood swelling by differential scanning calometry, found that the reduction of beech wood swelling cannot be attributed just to the disappearance of adsorption sites that go with hemicelluloses destruction. It is suggested that other phenomena, such as structural modifications and chemical changes of lignin, also play an important role.

And the impregnation of wood with aqueous solutions containing phosphoric acid followed by curing at 70 °C to 100 °C brought about the reaction between the impregnated chemical and the cellulose of wood. Wood impregnated by ammonium phosphates reported increased shear strength, and its swelling was reduced by up to 40% (Goldstein 1955).

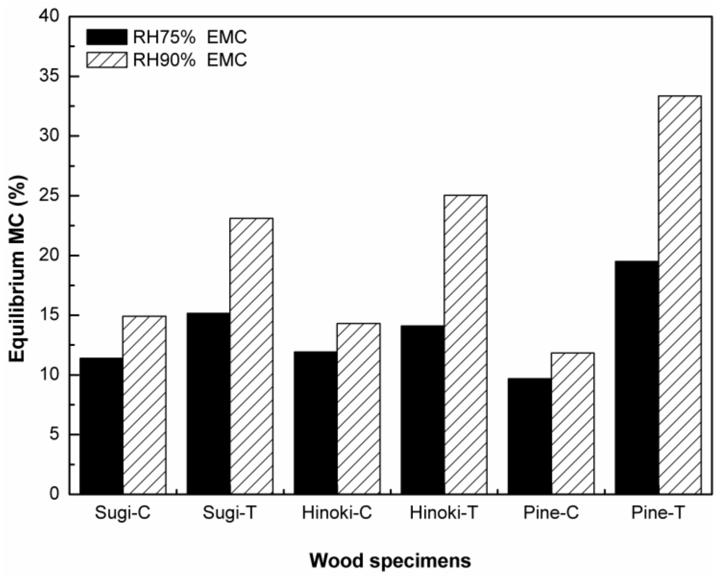

Fig. 2. EMC of control and treatment samples at 75% RH and 90% RH

Bulk coefficient, anti-swelling (ASE)

In order to further illustrate the anti-swelling of wood with the fire-retardant treatment, specimens with different uptake levels were prepared. The bulk coefficient and ASE were measured at 90% RH, and the uptake levels of Sugi, Hinoki, and Korean pine are shown in Table 3. Compared with the controls, the bulk coefficient of three wood were lowered after the fire-retardant treatment. The B% value of Sugi was minimally higher than that of Hinoki and Korean pine. This result might have been attributed to good permeability of Sugi. The EMC increased with the fire retardant treatment, while swelling decreased. The antiswelling efficiency (ASE) values were calculated on a volumetric basis and represent the swelling that flame retardant treatment prevents when compared to the swelling of a control specimen. It was clear that the treated specimens displayed improved ASE values compared to untreated wood specimen (ASE=0). The ASE values were 66.5% for Sugi, 71.7% for Hinoki, and 58.6% for Korean pine, respectively. The results were similar to other studies on wood modification with different chemical treatment. Godstein (1955) reported that the ASE of furfurylated white Korean pine was in the range of 41% to 70% with the weight percent gain changed from 17% to 120%. Lande *et al.* (2004) found that the ASE of furfurylated wood ranged from 35% to 75% with a new catalyst system, and stated that the ASE seems to be consistent even though chemical formulations of furfuryl and wood species varied. Chu *et al.* (2017a) found that in fire retardant and heat (at 150 °C for 30 min) combination treatment samples (group A), the values of ASE in tangential and radial directions were 36.8% and 51.9%, respectively in the humid test environment. Their research confirmed that the dimensional stability of heat-treated wood was improved by N-P pre-treatment. Jiang *et al.* (2015) obtained ASE value of 52% by treatment of APPUF1.0 (nitrogen-phosphorous based flame retardant coupled with reactive urea-formaldehyde (UF) oligomer) with 20% concentration.

It was believed that the good dimensional stability of treated wood was due to a bulking effect. The amorphous region of wood cell wall was penetrated with flame retardant molecules during impregnation, which resulted in the enlargement of space distance among microfibrils and reducing the bulky inflation after water immersion or moisture sorption. Moreover, the decreased hydrophilicity might result from the breaking down of hemicelluloses, modification of lignin, redistribution of wood extractives, and a decreasing number of hydroxyl groups in the wood cell walls.

Table 3. Dimensional Stability Evaluation of Sugi, Hinoki, and Korean Pine

Samples	Uptake (g/cm^3)	N	B (%)	MC (%)	ASE (%)
S-C	0	20	3.25 (0.73)	12.81 (0.23)	0
S-T	0.135	20	1.09 (0.37)	19.95 (2.64)	66.5
H-C	0	20	2.99 (1.32)	12.01 (0.20)	0
H-T	0.195	20	0.84 (0.55)	14.50 (0.86)	71.7
K-C	0	20	2.90 (1.06)	11.09 (0.18)	0
K-T	0.201	20	1.20 (0.99)	15.64 (0.54)	58.6

Note: Numbers in parenthesis are standard deviations; S, Sugi; H, Hinoki; K, Korean pine; B%, bulk coefficient; MC%, moisture content; ASE, anti-swelling efficiency

Hygroscopic behavior: Bulk coefficient of water immersion

The results on the bulking coefficient (B%) after water immersion for the continuous 7-day period are shown in Fig. 3. An almost maximum swelling was achieved after 1 day of soaking for all of the specimens. Afterward, the swelling rate was almost stable. The specimens with treatment were more dimensionally stable than the controls. This result further confirmed that the fire-retardant treatment increased the dimensional stability of wood by decreasing the volume change. The B% of the control specimen was higher than that of the specimens with the fire-retardant treatment, regardless of wood species. Compared with controls, the BC% of the specimens with treatment were decreased 45%, 86%, and 38% for Sugi, Hinoki, and Korean pine, respectively. The results were consisted with other research from Chu *et al.* (2017a). They found that the volumetric swelling of group A was 51.4% in the water bath.

This implied that flame retardant were deposited extensively into wood cell walls and thus were effective in reducing the swelling of wood specimens during water immersion. It was speculated that the impregnated flame retardant molecules could block sorption sites in the wood cell through reactions with free hydroxyl group from polysaccharides, forming cross-linking network between wood cell wall and flame retardant. As expected, the improvement of B% value was also related to increasing of ASE.

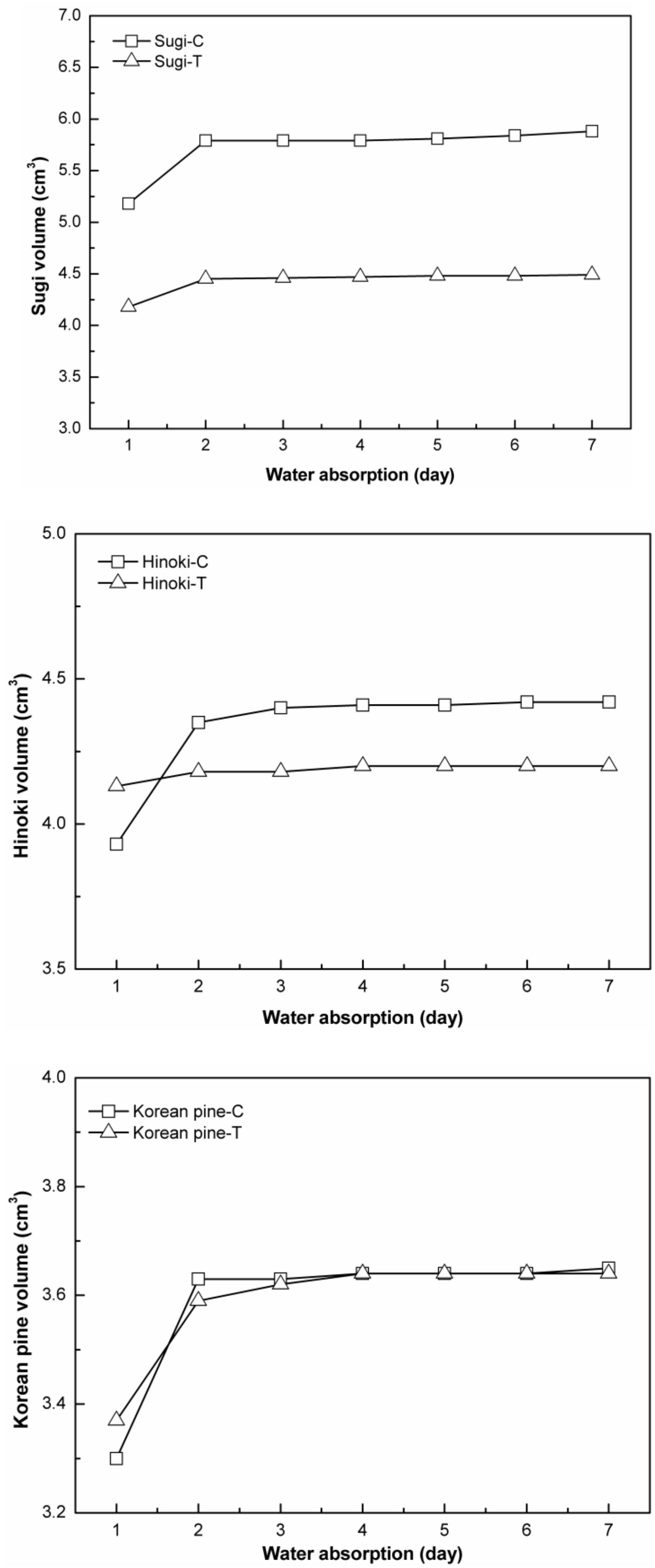

Fig. 3. Volume change of Sugi, Hinoki, and Korean pine samples with and without treatment as a function of water absorption time

Color Variation before and after Fire-retardant Treatment

Color has also been studied as a nondestructive criterion for monitoring changes in other wood properties of wood. Table 4 presents the results of the colorimeter variables. The total color difference (Δ*E*) was employed to measure the effect of the fire retardant impregnation on the wood samples.

It was found that coordinate *a** shifted negatively from 32.7 to 27.6, which meant that the board became greener. And coordinate *b** shifted negatively from 33.2 to 27.2, which indicated a loss of the yellow color. The coordinate *L** describes the lightness between 0 and 100, and the decrease in *L** meant a shift to darkness after the treatment.

The final color of the wood samples (*E**), representing the overall color changes of the samples in comparison to corresponding samples before treatment, was noticeably changed as well as the color saturation Δ*C*. The fire-retardant treatment was shown to modify the surface color. The overall Δ*E* was 40, which was similar to other research results of wood with a heat treatment. The decrease in color saturation (*C**) values was mainly attributed to the changes of the *a** and *b** values, due to the fire-retardant treatment. Wang *et al.* (2005) reported that the Δ*E* of red pine was 12 after treatment with guanylurea phosphate (GUP) and boric acid fire retardant. Bekhta and Niemz (2003) found that the total color difference increased from 22 to 28% after 2 h of treatment to the range 43 to 57% after 24 h of treatment. They concluded that heat treatment causes color changes results in high reduction in the hemicellulose content, and is thus an improvement of the dimensional stability of the wood. Matsuo *et al.* (2014) investigated color changes in Keyaki and Sugi wood with heat treatment at 90 to 180 °C. After 4 h heat treatment Δ*E* was about 20 to 40. It was believed that thermal oxidation causes color changes during both natural aging and heat treatment.

The decrease of luminance on the wood surface could be explained by the formation of hemicelluloses and extractive thermal degradation products or possibly attributed to lignin polymerization reactions after the fire-retardant treatment. According to Chow and Mukai (1972), the darkness is a result of the change in the α-cellulose color that is promoted probably by oxidation reactions. Del Menezzi *et al*. (2009) argued that these reactions also change the color of other compounds and that extractive migration has an important role during thermal treatment. Considering wood samples underwent fire-retardant treatment and a drying process, it is considered that the phosphate acid released from the APP and GUP further accelerate the degradation of hemicellulose and the migration of extractives after fire retardant treatment during the drying process with high temperature. Consequently, it was inevitable to result in color change.

Table 4. Color Variation of Specimens before and after Treatment

Specimen	N	Color parameter				
		L *	*a**	*b**	*C**	Δ*E*
Before treatment	20	42.9 (6.7)	32.7 (5.3)	33.2 (6.1)	191	40
After treatment	20	36.1 (5.6)	27.6 (4.5)	27.2 (4.9)	146	
Calculation	40	6.8	5.1	6	45	

Note: Numbers in parenthesis are standard deviations

CONCLUSIONS

1. The EMC of Sugi, Hinoki, and Korean pine all increased for 75% RH and 90% RH at 40 °C. However, the results of the specimens after treatment were lower than that of the controls for the radial and tangential swelling of 1% MC increments, presenting higher dimensional stability.
2. The determination of the bulking coefficient (BC%) after water immersion for the continuous 7 day period further confirmed that the fire-retardant treatment increased the dimensional stability of wood. The results were quite similar with the results from some other research which confirmed that the dimensional stability of heat-treated wood was improved by N-P fire retardant pre-treatment.
3. Color measurements of the nitrogen-phosphorus fire-retardant chemical-treated specimens revealed a decrease in L^*, decrease in a^* and b^* parameter values, and the color saturation C^* (the ΔE is 40) of the samples as well. These changes indicated the tendency of wood surfaces to darken compared with wood specimens before fire-retardant treatment. This result is consisted with other N-P fire retardant and heat treatment methods. And these treatments inevitably have adverse effects on wood color.
4. As a whole, despite the undesirable color change, good dimensional stability and flame retardant of N-P fire retardant wood will provide much wider application for Sugi, Hinoki, and Korean pine in residential and non-residential construction and building field. This study provides a kind of fire retardant that is low cost and shows excellent fire-retardant effect for the practical application.

ACKNOWLEDGMENTS

The authors are grateful for the support of the National Natural Science Foundation of China (31700483) and Doctoral Scientific Research Foundation of Beihua University (101416034). Thanks are also given to the National Natural Science Foundation of China (31470022), Special plan for economic structure strategic adjustment in Jilin Province (2015Y079), and National Public Welfare Special Foundation (201504502).

REFERENCES CITED

ASTM D2244-07 (2007), "Standard practice for calculation of color tolerances and color differences from instrumentally measured color coordinates," ASTM International, West Conshohocken, PA.

Bekhta, P., and Niemz, P. (2003). "Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood," *Holzforschung* 57(5), 539-546. DOI: 10.1515/hf.2003.080

Cai, X. L., Riedl, B., Zhang, S. Y., and Wan, H. (2007). "Effects of nanofillers on water resistance and dimensional stability of solid wood modified by melamine-urea-formaldehyde resin," *Wood Fiber Sci.* 39(2), 307-318.

Chow, S. Z., and Mukai, H. N. (1972) "Effect of thermal degradation of cellulose on wood-polymer bonding," *Wood Sci.* 4(4), 202-208.

Chu, D., Mu, J., Zhang, L., and Li, Y. (2017a). "Promotion effect of N-P fire retardant pre-treatment on heat-treated poplar wood. Part 1: Color generation, dimensional stability, and fire retardancy," *Holzforschung* 71(3), 207-215. DOI: 10.1515/hf-2016-0082

Chu, D., Mu, J., Zhang, L., and Li, Y. (2017b). "Promotion effect of N-P fire retardant pre-treatment on heat-treated poplar wood. Part 2: Hygroscopicity, leaching resistance, and thermal stability," *Holzforschung* 71(3), 217-223. DOI: 10.1515/hf-2016-0213

del Menezzi, C. H. S., Tomaselli, I., Okino, E. Y. A., Teixeira, D. E., and Santana, M. A. E. (2009). "Thermal modification of consolidated oriented strandboards: Effects on dimensional stability, mechanical properties, chemical composition and surface color," *Eur. J. Wood Wood Prod.* 67(4), 383-396. DOI: 10.1007/s00107-009-0332-2

Dubey, M. K., Pang, S., and Walker, J. (2012). "Changes in chemistry, color, dimensional stability and fungal resistance of *Pinus radiata* D. Don wood with oil heat-treatment," *Holzforschung* 66(1), 49-57. DOI: 10.1515/hf.2011.117

Gao, M., Ling, B., Yang, S., and Zhao, M. (2005). "Flame retardance of wood treated with guanidine compounds characterized by thermal degradation behavior," *J. Anal. Appl. Pyrol.* 73(1), 151-156. DOI: 10.1016/j.jaap.2005.01.006

Goldstein, I. S. (1955) "The impregnation of wood to impart resistance to alkali and acid," *Forest Prod. J.* 5(4), 265-267

Huo, S., Wang, J., Yang, S., Wang, J., Zhang, B., Zhang, B., Chen, X., and Tang, Y. (2016). "Synthesis of a novel phosphorus-nitrogen type flame retardant composed of maleimide, triazine-trione, and phosphaphenanthrene and its flame retardant effect on epoxy resin," *Polym. Degrad. Stabil.* 131, 106-113. DOI: 10.1016/j.polymdegradstab.2016.07.013

Jiang, J., Li, J., and Gao, Q. (2015). "Effect of flame retardant treatment on dimensional stability and thermal degradation of wood," *Constr. Build. Mater.* 75, 74-81. DOI: 10.1016/j.conbuildmat.2014.10.037

Jin, E., and Chung, Y. J. (2016). "Combustion characteristics of wood panels treated with phosphorus-nitrogen additives," *BioResources* 11(2), 4319-4331. DOI: 10.15376/biores.11.2.4319-4331

Kamperidou, V., Barboutis, I., and Vasileiou, V. (2013). "Response of colour and hygroscopic properties of Scots pine wood to thermal treatment," *J. Forest Res.-JPN* 24(3), 571-575. DOI: 10.1007/s11676-013-0389-y

Kang, C. W., Wen, M. Y., Park, H. J., Kang, H. Y., Kang, S. G., and Matsumura, J. (2017). "Changes in some mechanical and physical properties and anatomical structure of spruce and larch wood after fire-retardant treatment," *BioResources* 12(1), 1358-1368. DOI: 10.15376/biores.12.1.1358-1368

Lande, S., Westin, M., and Schneider, M. (2004). "Properties of furfurylated wood," *Scand. J. Forest Res.* 19(Sup 5), 22-30. DOI: 10.1080/0282758041001915

Li, J. Z., Furuno, T., and Katoh, S. (2000). "Dimensional stability and flame resistance of silicate-acetylated and -propionylated wood composites," *J. Wood Chem. Technol.* 20(4), 441-453. DOI: 10.1080/02773810009351893

Lowden, L. A., and Hull, T. R. (2013). "Flammability behaviour of wood and a review of the methods for its reduction," *Fire Science Reviews* 2(1), 4. DOI: 10.1186/2193-0414-2-4.

Matsuo, M., Umemura, K., and Kawai, S. (2014). "Kinetic analysis of color changes in keyaki (*Zelkova serrata*) and sugi (*Cryptomeria japonica*) wood during heat treatment," *Journal of Wood Science* 60(1), 12-20. DOI: 10.1007/s10086-013-1369-8

Pan, J., Mu, J., Wu, Z., and Zhang, X. (2014). "Effect of nitrogen-phosphorus fire retardant blended with $Mg(OH)_2$/$Al(OH)_3$ and nano-SiO_2 on fire-retardant behavior and hygroscopicity of poplar," *Fire Mater.* 38(8), 817-826. DOI: 10.1002/fam.2224

Park, H. J., Wen, M. Y., Cheon, S. H., Kang, C. W., and Matsumura, J. (2015). "Fire retardant performance and thermal degradation of Korean pine treated with fire retardant chemical," *J. Fac. Agr. Kyushu U.* 60(1), 183-189.

Park, H. J., Wen, M. Y., Kang, C. W., and Sun, Y. X. (2017). "Development of physical pretreatment method for wood fire retardant impregnation," *BioResources* 12(2), 3778-3789. DOI: 10.15376/biores.12.2.3778-3789

Repellin, V., and Guyonnet, R. (2005). "Evaluation of heat-treated wood swelling by differential scanning calorimetry in relation to chemical composition," *Holzforschung* 59(1), 28-34. DOI: 10.1515/HF.2005.005

Wang, Q., Wang, W., and Winandy, J. E. (2005). "Effect of new GUP-B fire retardant on mechanical properties of Korean pine when exposed to elevated temperature," *Forest Prod. J.* 55(12), 214-220.

Wen, M. Y., Kang, C. W., and Park, H. J. (2014). "Impregnation and mechanical properties of three softwoods treated with a new fire retardant chemical," *J. Wood Sci.* 60(5), 367-375. DOI: 10.1007/s10086-014-1408-0

Article submitted: July 16, 2017; Peer review completed: September 10, 2017; Revised version received and accepted: November 21, 2017; Published: December 1, 2017.
DOI: 10.15376/biores.13.1.764-775