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Impregnation and mechanical properties of three softwoods treated with a new fire retardant chemical

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Abstract Three softwoods, Sugi (*Cryptomeria japonica*), Korean Pine (*Pinus koraiensis*) and Hinoki (*Chamaecyparis obtusa*), were vacuum–pressure impregnated with a fire retardant chemical consisting of ammonium phosphate polymer (APP), guanyl urea phosphate (GUP), phosphonic acid and a minor amount of additives. The variation in impregnation between and within wood species was investigated. A significant relationship and similar trends were found between fire retardant chemical (FR) uptake and specific gravity (SG), as well as void volume filled (VVF) and SG. Moreover, the effects of fire retardant treatment on mechanical properties, including modulus of rupture (MOR), static modulus of elasticity (MOE) and dynamic modulus of elasticity (DMOE), were evaluated. The results indicated that the trend of impregnation and regression function varied between species and positions within the same species. However, the relationship of SG and chemical uptake and that of VVF and chemical uptake could be represented by a positive linear regression, and the trends were similar between wood species. Both of SG and VVF increased with increasing FR uptake. After fire retardant treatment, the MOR and static MOE were reduced compared with before treatment. Conversely, the DMOE increased after treatment.

Keywords Fire retardant chemical impregnation · Void volume filled · Chemical uptake · Mechanical properties

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Introduction

Woods can provide good service under proper use conditions. However, under unfavorable conditions, they may easily be damaged and destroyed by fire. The treatment of wood with various solutions is applied to improve wood or to chemically convert wood into a different products [1].The proper application of fire retardant chemicals can protect wood from fire, thus prolonging the service life of wood. Effectiveness of impregnation treatment depends on the chemical formulation selected, method of application, proportion of sapwood to heartwood, moisture content of the wood, amount of chemical retained and depth of chemical penetration and distribution [2–5].

Different position and anatomical factors combine to respond for the variation in chemical impregnation treatability. During the chemical treatment of wood, it is necessary to impregnate chemicals into the wood uniformly and deeply, which depends on permeability of wood. The difficulty of liquid flow between cell lumens varies among wood species and wood portions. Sapwood of the most commercial species can be impregnated more easily than heartwood. Impregnation of heartwood is generally limited by pits clogged with debris that makes them resistant to fluid flow and limits the effectiveness fire retardant treatment. Due to the anatomical structure of wood, an intercapillary network makes it possible to impregnate wood more or less thoroughly [6]. Since wood is an uneven complex porous material, its permeability varied widely. The variability of fluid permeability of wood is much greater than other properties and there exist great differences among different families, genera and tree species, even within parts of the same tree. Therefore, the wood fire retardant treatment process is closely related to wood permeability.

Many studies have been conducted on the effects of various treatments on the mechanical properties of wood. Researchers have recognized that fire retardant treatment and post re-drying reduced the initial strength properties of wood. Furthermore, it is reported that pressure treatment caused a decrease of 8–10 % in bending strength of different wood types [7]. Other studies reported that salty impregnation materials increased the compression strength by 4.6–9.6 %, but decreased the bending strength by 2.9–16 % [8, 9]. This reduction in strength occurs when wood products are exposed to the elevated temperatures that are often experienced as a result of solar loads in roof applications. Some research has investigated how much and why fire retardant affected the properties of treated wood [10, 11].

To better understand the treatability on softwoods with the new fire retardant chemical and to help process fire retardant treatment, it is important to know the impregnation treatability between and within three softwoods as well as the impregnation effect on the mechanical properties of the treated softwoods. The object of this study is on one hand to investigate the impregnation treatability with a new fire retardant chemical, with ammonium phosphate polymer (APP), guanyl urea phosphate (GUP) and phosphonic acid as main components, into three softwoods mostly used in Korea, including Sugi (*Cryptomeria japonica*), Korean Pine (*Pinus koraiensis*) and Hinoki (*Chamaecyparis obtusa*) by a conventional vacuum–pressure method. One the other hand, fire retardant treatment effect on mechanical properties of the woods was examined, in attempt to provide a reasonable treatment process for better application of the fire retardant chemicals, and thus get the desired fire retardant properties.

Materials and methods

Softwood species

Three softwood species, including Sugi (*Cryptomeria japonica*), Korean Pine (*Pinus koraiensis*) and Hinoki (*Chamaecyparis obtusa*), were selected for this study. Defect-free sapwood, sap-heart wood and heartwood of each species with the dimensions of 910 (longitudinal) × 100 (tangential) × 10 (radial) mm with similar weight were selected. Prior to treatment, boards were kiln-dried to 8 % moisture content (MC), and then labeled and weighed.

Fire retardant chemicals

The main components of water soluble fire retardant solution are ammonium phosphate polymer (APP), guanyl urea phosphate (GUP), phosphonic acid, acrylamide-acrylic acid-*N*-{3-(dimethylamino)propyl} methacrylamide copolymer, 2-benzisothiazolin-3-one and a minor amount of additives, with a 25 % concentration of the fire retardant, a specific gravity of 1.13 (20 ± 2 °C) and a pH of 7.6 (20 ± 2 °C).

Treatment of softwood with fire retardant chemical

Vacuum–pressure impregnation process

Wood samples were randomly placed in a vertical cylindrical vessel. A vacuum of −0.098 MPa was applied for 10 min for removing the air contained within wood. When the vacuum was released, the fire retardant chemicals refluxed into the vessel, and the pressure was raised to a given pressure of 10, 15 and 20 kgf/cm^2, respectively. The pressure condition was maintained for a given time. Then pressure was released, and samples were carried out and weighed. The mass increases were determined from the mass difference between pre-treated and treated boards. The pressure process was repeated as above with a 3-min interval in the beginning 30, 10 min interval after 30 min, and total pressuring time was 180 min. After impregnation, the samples were air-dried for 2 weeks and then dried at 60 °C to a MC of 12 %.

Chemical uptake

The amount of FR chemical absorbed by the wood sample was calculated by uptake as follows: uptake (g/cm^3) = $(M_t - M_u)/V$, where $(M_t - M_u)$ is mass (gram) of FR chemical solution absorbed, and V is the volume of sample in cm^3.

Specific gravity

After treatment, the boards with different FR uptake were cut into 50 × 50 × 10 mm samples, oven-dried at 103 °C (±2 °C) until no mass change was observed and cooled down in the desiccators. Then the length, width and thickness were measured with a micrometer (accurate to ±0.1). The specific gravity of the samples was calculated based on the oven-dry mass and green volume.

Void volume filled (VVF)

Based on the assumption that, within a given volume of the sample with a known basic density and constant cell wall density (1500 kg/m^3), there will be a void volume basically composed of cell lumina, intercellular spaces, and penetrable cell wall voids, which can be filled by liquids [12, 13]. Treatability in this study is also expressed as the ratio of fire retardant chemical uptake to the potential volume that could be occupied if the samples were completely filled. The porosity as void volume (P) and VVF of wood samples were calculated as follows:

$$P = \left(1 - \frac{SG_t}{1.50}\right) \times 100 \quad (1)$$

$$VVF = \left(\frac{M_t - M_u}{V \times P}\right) \times 100 \quad (2)$$

where SG_t is the specific gravity of the treated sample, M_t (g) is the mass of treated sample, and M_u (g) is the mass of the untreated sample. V (cm^3) is the volume of sample before oven drying with 12 % MC.

Mechanical properties

The sample size was 32 (length) × 2 (width) × 1 (thickness) cm with 20 samples for each species. The static bending tests were conducted in accordance with the third-point loading method for lumber and center loading method for a specimen, using a Shimadzu autograph universal testing machine. All the specimens loaded in the flat direction (wise loading) for the bending tests. The span was 140 cm. The proportional limit, ultimate load, and deflection were obtained from load–deflection curves, and the static MOE and the MOR were calculated.

DMOE in the longitudinal direction was estimated from the transverse vibration of the small beam with free–free conditions. The specimen was supported by two strings, as shown Fig. 1. Supporting positions were 0.224 of total length from both ends. These positions are a nodal point for the first mode of this free vibration. On one of the beam ends, the impulse hammer tapped the specimen, and the accelerometer detected the signal from the center of the beam. The multichannel signal analyzer received these two signals simultaneously, and the frequency response function curve between impulse hammer and transducer was achieved. From the spectral analysis shown in Fig. 1, the resonance frequency of transverse vibration was estimated, and the DMOE was calculated using the following formulae. Here, the influence of concentration mass by accelerometer and shear stress was not considered.

$$DMOE = 48\pi^2 \rho l^4 f^2 /\ m^4 h^2 \quad (3)$$

where $DMOE$ = dynamic modulus of elasticity, f = resonance frequency, L = length of the specimen, ρ = density of the specimens, h = thickness, and m is a constant (4.73 for the fundamental mode of vibration).

Data analysis

Impregnation, chemical uptake and mechanical data were analyzed using the statistical software IBM SPSS Statistics (SPSS 19.0). To determine whether there were any significant differences in mechanical properties before and after treatment, the mechanical data were compared by a two-tailed group t test with the levels set at 1 %.

Results and discussion

Treatability of wood with new fire retardant chemicals

Flame retardant uptake as function of impregnation time soft for wood

Figures 2, 3, and 4 show the experimental data and model impregnation curves obtained with sapwood, sap-heart

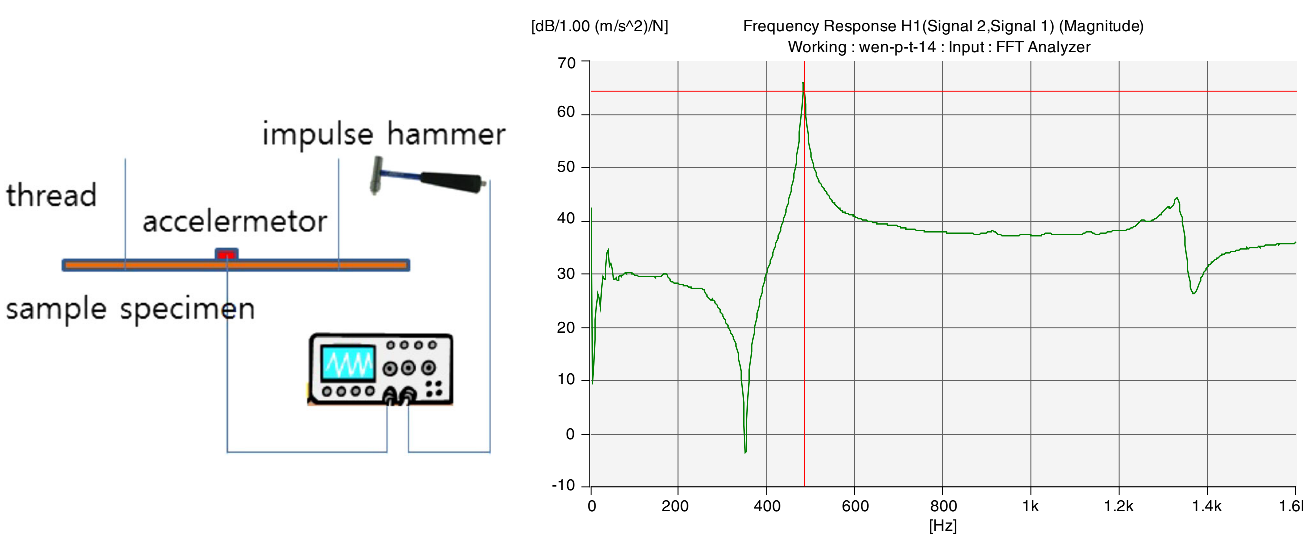

Fig. 1 Schematic diagram for dynamic modulus elasticity (DMOE) measuring apparatus and typical frequency response function curve

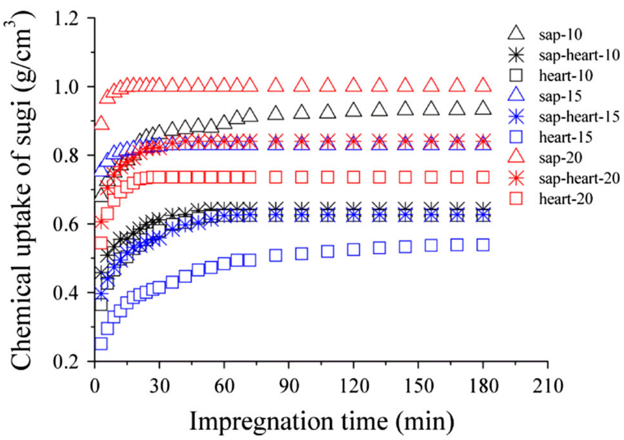

Fig. 2 Chemical uptake of Sugi sapwood, sap/heart wood and heart wood as function of impregnation time

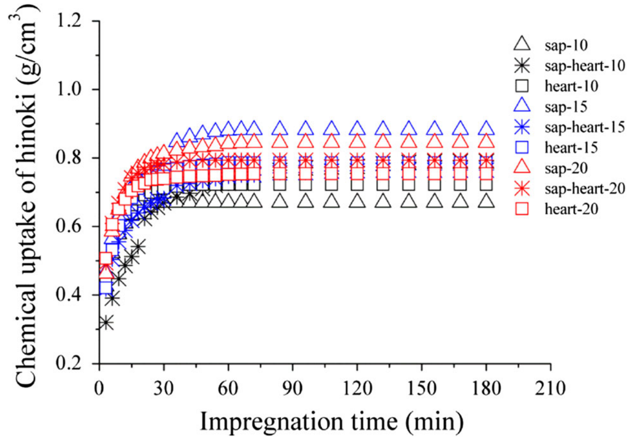

Fig. 4 Chemical uptake of Hinoki sapwood, sap/heart -wood and heart wood as function of impregnation time

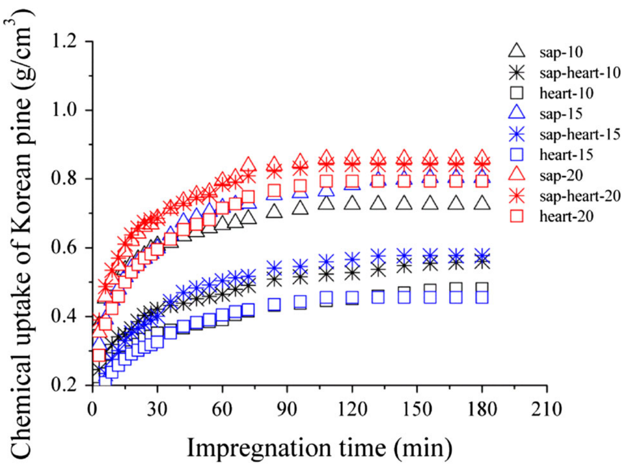

Fig. 3 Chemical uptake of Korean pine sapwood, sap/heart wood and heart wood as function of impregnation time

wood and heartwood of Sugi, Korean pine and Hinoki at fixed pressures of 10, 15 and 20 kgf/cm^2. Models describing the relationship between treatment time and impregnation treatability can be used to develop more controlled treatments. The exponential function of chemical uptake as a function of treatment time for three wood species with different positions was fitted, as shown in Table 1.

From the impregnation curves, it can be observed that impregnation treatability varied between species and within species (sapwood, sap-heart wood and heartwood), as well as pressure level and time. We found that a significant initial increase occurred in all curves as shown in Figs. 2, 3, and 4, especially for the sapwood of Sugi, and then a progressive trend of uptake increase continued as modeled by exponent regression. The regression has an excellent exponent fit, whereas different species and positions have divergent fits models. Furthermore, as expected, sapwood is most easily impregnated, followed by sap/heartwood, and heartwood exhibited inferior impregnation. This is attributed to the anatomy variations in positions within species. It is reported that the variation in permeability among sapwood, intermediate wood and heartwood was caused by bordered pit aspiration, which occurs during heartwood formation [14].

Wood anatomical structure is related to physical properties of wood, and the states of bordered pit pairs affect the permeability of softwoods. The permeability is shown by the magnitude of bulk flow of fluids through wood, mainly through the cell limens and pits. Pits varied among different wood species, thereafter and this affected the impregnation treatability. The impregnation treatability is closely related to the permeability and penetrability of wood species as wood possesses a capillary structure, which provided primary paths for liquid penetration into wood [15]. As a capillary porous medium, wood pore structure is determined by interconnected cell lumen and cell wall openings (pits). Larger and more numerous, the pit membrane openings produce higher permeability [16]. Capillary structures, consisting mainly of tracheids in softwoods, as well as ray cells, resin canal and pit membranes, play an important role in liquid penetration into wood. Sapwood contains tracheids, vessels and living parenchyma. Whereas, heartwood is physiologically inactive contains a large amount of resinous and phenolic extractives with biotic resistance. One of the main reasons for obstruction of liquid flow in heartwood is believed to be closure of the bordered pits by pit aspiration and occlusion. Additionally, the surfaces of the pit membranes of heartwood are frequently covered with heartwood extractives, especially for pinewood [17]. The open pits in tracheids and tracheid length together determine the permeability, and consequently determine the solution uptake [18].

Table 1 Regression function of chemical uptake as a function of time for different species, positions and pressure

Species	Pressure	Position	Regression function	R^2
Sugi	10	Sap	0.922 × (1 − exp (− exp (−3.067) × (Time + 26.664)))	0.986
		Sap-heart	0.644 × (1 − exp (− exp (−2.732) × (Time + 16.992)))	0.992
		Heart	0.625 × (1 − exp (− exp (−2.347) × (Time + 11.359)))	0.994
	15	Sap	0.829 × (1 − exp (− exp (−1.993) × (Time + 14.809)))	0.980
		Sap-heart	0.629 × (1 − exp (− exp (−2.983) × (Time + 17.574)))	0.993
		Heart	0.534 × (1 − exp (− exp (−3.501) × (Time + 21.273)))	0.989
	20	Sap	(1 − exp (− exp (1.049) × (Time + 3.289)))	0.997
		Sap-heart	0.840 × (1 − exp (− exp (−2.129) × (Time + 8.269)))	0.986
		Heart	0.737 × (1 − exp (− exp (−1.759) × (Time + 4.971)))	0.997
Korean pine	10	Sap	0.723 × (1 − exp (− exp (−3.373) × (Time + 22.235)))	0.982
		Sap-heart	0.553 × (1 − exp (− exp (−3.752) × (Time + 26.805)))	0.984
		Heart	0.475 × (1 − exp (− exp (−3.760) × (Time + 23.502)))	0.983
	15	Sap	0.794 × (1 − exp (− exp (−3.447) × (Time + 15.647)))	0.991
		Sap-heart	0.576 × (1 − exp (− exp (−3.484) × (Time + 11.077)))	0.994
		Heart	0.460 × (1 − exp (− exp (−3.531) × (Time + 14.480)))	0.994
	20	Sap	0.857 × (1 − exp (− exp (−3.280) × (Time + 13.795)))	0.987
		Sap-heart	0.838 × (1 − exp (− exp (−3.254) × (Time + 16.170)))	0.983
		Heart	0.796 × (1 − exp (− exp (−3.428) × (Time + 13.498)))	0.991
Hinoki	10	Sap	0.671 × (1 − exp (− exp (−1.801) × (Time + 3.083)))	0.997
		Sap-heart	0.777 × (1 − exp (− exp (−3.017) × (Time + 8.204)))	0.988
		Heart	0.722 × (1 − exp (− exp (−2.006) × (Time + 4.061)))	0.998
	15	Sap	0.883 × (1 − exp (− exp (−2.673) × (Time + 8.399)))	0.995
		Sap-heart	0.759 × (1 − exp (− exp (−2.769) × (Time + 10.819)))	0.991
		Heart	0.784 × (1 − exp (− exp (−2.268) × (Time + 4.688)))	0.996
	20	Sap	0.841 × (1 − exp (− exp (−2.233) × (Time + 4.691)))	0.996
		Sap-heart	0.793 × (1 − exp (− exp (−1.953) × (Time + 3.997)))	0.998
		Heart	0.751 × (1 − exp (− exp (−1.982) × (Time + 5.361)))	0.994

In addition, there appear to be species differences in impregnation treatability of the softwoods. The impregnation curves demonstrated that the chemicals uptake initially increased dramatically and quickly approached the maximum chemical uptake at 30 min for Sugi, 60 min for Korean pine, and 30 min for Hinoki, respectively, regardless of position and impregnation pressure level. Sugi, which is extensively planted in Japan, has a low permeability. According to Matsumura J et al. [14], the percentage of aspirated pits in heartwood of air-dried Sugi is 65–80 %. However, the chemical uptake trends of Hinoki are similar to each other among sap, sap/heart and heart wood. The variance in chemical uptake by position also existed in Korean pine, but the uptake increased only mildly as treatment time increased. In terms of Sugi, however, when impregnation pressure level was increased from 10 to 15 to 20 kgf/cm^2, the uptake increased by 4–17 % for sapwood, 10–47 % for sap/heartwood and 40–47 % for heartwood. In the case of Korean pine, the uptake increased by 7.5–19 % for sapwood, 8–69 % for sap/heart wood and 3–83 % for heartwood. However, for Hinoki, the uptake increased by 19–21 %, 2–17 % and 4–5 % for sapwood, sap/heart wood and heartwood, respectively. Therefore, for Sugi and Korean pine, if more chemical uptake is desired, it is important to increase pressure level. In contrast, there is no significant difference in the uptake of Hinoki, regardless of position when increasing pressure level as the trends in Fig. 4 exhibited.

Relationship between specific gravity and chemical uptake

A significant positive correlation was found between the SG and chemical uptake (UT), and VVF and chemical uptake of specimens as shown in Figs. 5 and 6. Their relationship can be represented by positive linear regression formulas as follows:.

Sugi $SG = 0.234 + 0.369UT.\ R^2 = 0.928^*$
Pine $SG = 0.318 + 0.330UT.\ R^2 = 0.903^*$
Hinoki $SG = 0.384 + 0.334UT.\ R^2 = 0.872^*$

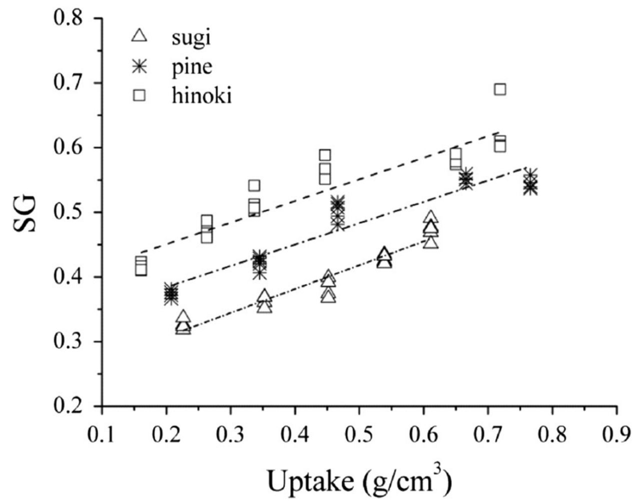

Fig. 5 Relationship between specific gravity of treated wood and chemical uptake

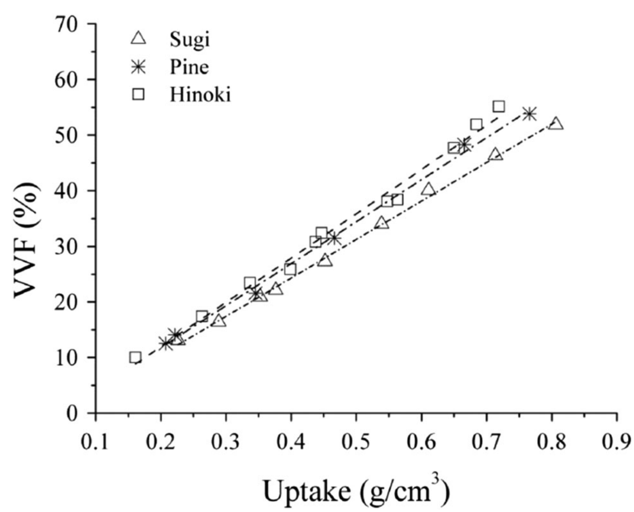

Fig. 6 Relationship between void volume filled of treated wood and chemical uptake

Sugi $VVF = -3.353 + 69.167UT.\ R^2 = 0.998*$
Pine $VVF = -0.330 + 75.526UT.\ R^2 = 0.997*$
Hinoki $VVF = -3.962 + 79.627UT.\ R^2 = 0.990*$

The fire retardant chemical penetrates deep into the wood by vacuum–pressure methods. When impregnated, the structure of wood is viewed as being similar to that of a sponge, with cell cavities and cell walls. The aim of fire retardant treatment is to coat these walls with fire retardant chemicals to protect the structure from fire. First, the vacuum removes the air from the cavities to create space for the fire retardant solution, which is then forced deep into the wood under pressure. Treatability in this study is also expressed as the ratio of fire retardant chemicals to the potential volume that could be occupied if the samples were completely occupied. Furthermore, the fire retardant performance is influenced significantly by the fire retardant chemical uptake. The chemical uptake is affected by SG. The SGs of Sugi, Korean pine and Hiniki were 0.31, 0.33 and 0.38 g/cm^3, respectively, before treatment. When the specimens were impregnated, the SG increased with the uptake increase, as shown in Fig. 5.

A lower specific gravity means fewer cell wall materials, which is analogous to higher void volume and facilitates absorption of a greater amount of chemicals. Therefore, Sugi absorbed more chemicals due to its lower SG level. At the same chemical uptake level, Hinoki presented higher SG and void volume-filled percentage. Moreover, despite different anatomical features and permeability, the trends of SG increased with uptake and are similar among three species with similar slopes as shown in the expressions.

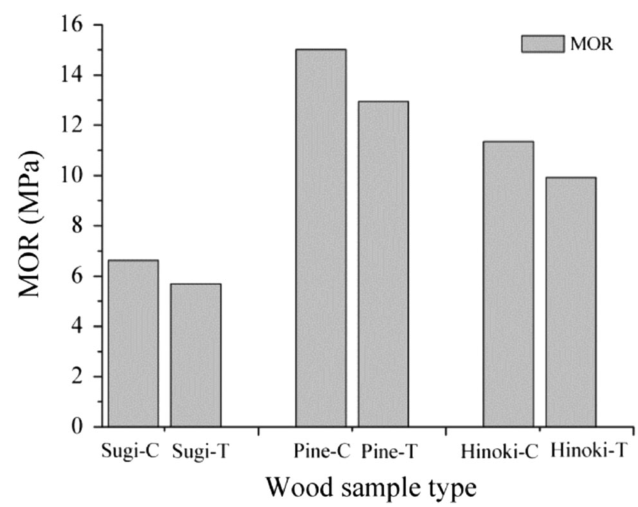

Fig. 7 Effect of impregnation treatment on MOR

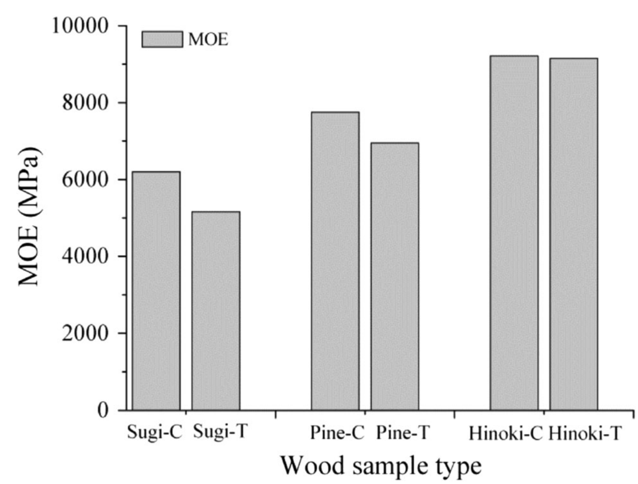

Fig. 8 Effect of impregnation treatment on MOE

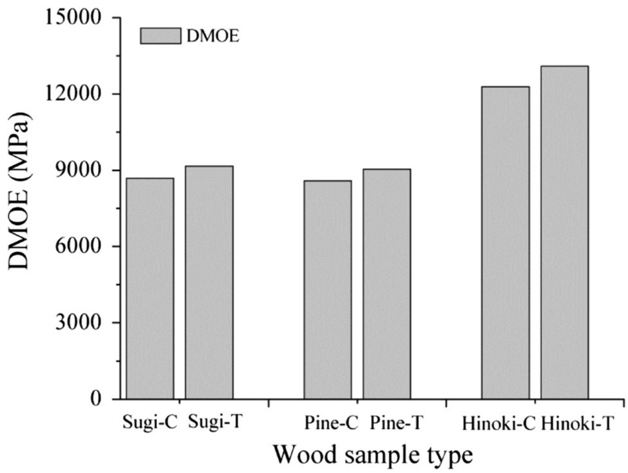

Fig. 9 Effect of impregnation treatment on DMOE

Mechanical properties

The MOR, static MOE and DMOE, are the most common properties used to indicate the quality of wood and they are very important factors in determining the strength of wood. The MOE, one of primary indexes in evaluating mechanical properties of wood, indicates the degree of wood-resisting distortion. A higher value of MOE indicates that the material is not easy to be distorted and has a high rigidity. In Figs. 7, 8, and 9, it can be observed that after treatment MOR and static MOE decreased by 12–14 % and 2–18 %, respectively; conversely, DMOE increased 3–5 %. By two-tailed group *t* test statistics as shown in Table 2, the differences in MOR, static MOE and DMOE were found statistically significant ($p < 0.01$) before and after treatment. Additionally, it was found that MOR was affected to a greater degree than static MOE. Similar trends can be seen with other fire retardant systems.

Phosphorus-based compounds are some of the best-known fire retardant treatments for wood. However, a significant problem with these compounds is the reduction in strength of treated wood products. Moreover, as previously reported by Winandy [10], those compounds have more significant negative effects on the viscoelastic properties than on the elastic properties of treated wood, as do other more acidic fire retardants. He noted that the physical and mechanical properties of wood are complex functions of cellular and polymeric structure and chemistry. He observed that changes in the chemical composition of wood directly corresponded to a loss of strength. Moreover, Wang [9] reported that a decrease in bending strength could be attributed to the fire retardant treatment resulting in a chemical component change in treated wood, especially in the hemicellulose content after drying. It is noted that, after treatment, the percentage of hemicelluloses decreased compared with the untreated specimens, and conversely, lignin residue increased for phosphoric acid-treated specimens that were kiln-dried after treatment. Drying of phosphoric acid-treated specimens caused some changes in wood components.

It is thought that the strength loss in wood might be closely related to the degradation of the branched units of hemicelluloses, while later advanced strength loss is related

Table 2 Effects of fire retardant treatment on the mechanical properties of Sugi, Korean pine and Hinoki

Species	Mechanical properties (MPa)	Treatment	*N*	*M* ± SD	*t*	Sig. (2-tailed)
Sugi	MOR	Control	18	6.64 ± 0.27	13.68	0.000
		Treatment	18	5.69 ± 0.19		
	MOE	Control	18	6316 ± 952	7.108	0.000
		Treatment	18	5162 ± 730		
	DMOE	Control	18	8840 ± 1240	−2.500	0.023
		Treatment	18	9160 ± 1280		
Pine	MOR	Control	16	11.35 ± 0.30	18.325	0.000
		Treatment	16	9.92 ± 0.34		
	MOE	Control	16	9309 ± 939	0.572	0.576
		Treatment	16	9154 ± 776		
	DMOE	Control	16	12300 ± 61700	−4.563	0.000
		Treatment	16	13100 ± 77000		
Hinoki	MOR	Control	18	14.96 ± 2.11	5.613	0.000
		Treatment	18	12.93 ± 1.30		
	MOE	Control	18	7750 ± 1232	7.726	0.000
		Treatment	18	6788 ± 1008		
	DMOE	Control	18	8610 ± 1790	−3.160	0.006
		Treatment	18	9040 ± 1089		

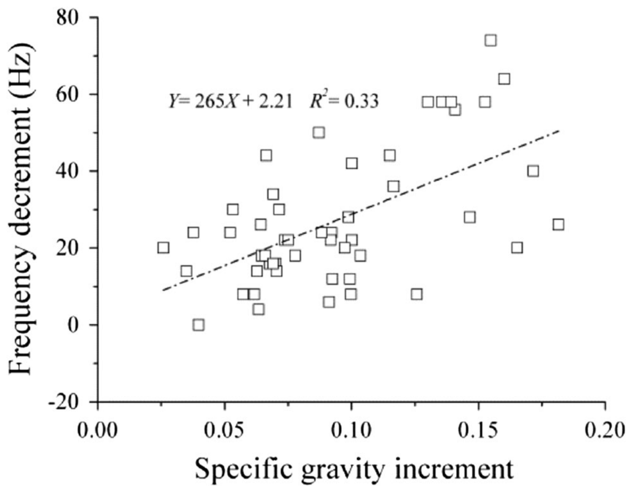

Fig. 10 The correlation between frequency decrement and specific gravity increment

to further degradation of residual hemicelluloses main chain and initial degradation of cellulose and lignin when wood underwent drying after impregnation.

In addition, the static MOE of the treated specimen decreased but DMOE was increased by the treatment. One reasonable explanation for this finding is that the differences of estimation range deformation by stress between the static MOE and DMOE. In the case of static MOE, the deformation range is bigger than DMOE. The other possible explanation is that, in terms of DMOE, the deformation range is restricted to a very small range so that the hardened treated wood surface causes an increase in DMOE. Moreover, the weight gain by impregnation caused an increase in density (ρ) of wood specimens but a decrease in resonance frequency (f). However, the decrease in resonance frequency ratio is smaller than the increase in density. The correlation between frequency decrement and density increment was shown in Fig. 10. Consequently, the increased DMOE values were obtained as calculated by expression (3).

Conclusion

Impregnation treatability and mechanical properties of three softwoods using the new developed fire retardant chemical were investigated for providing theory and technique basis for preparing fire retardant wood with proper process.

1. With this fire retardant, variance in chemical uptake by position existed in Sugi and Korea pine. When increasing pressure level, chemical uptake increase is most for Sugi, followed by Korea pine and least for Hinoki. Therefore, for Sugi and Korean pine, if more chemical uptake is desired, it is important to increase pressure level. In contrast, there is no significant difference in the uptake of Hinoki, regardless of position when increasing pressure level.
2. The SG and VVF of fire retardant treated woods increased with chemical uptake increasing. The relationship can be represented by a positive linear regression and the trends were similar between wood species. Furthermore, it was found that MOR, static MOE of three wood species decreased; conversely, DMOE increased as a result of fire retardant treatment.
3. In this study, thin board of the softwood was only used for impregnation. In the following study, research will be conducted on ridgepole impregnation, and if necessary combined with incising method, in an attempt to apply it to reconstruct traditional wooden structure. In addition, after fire retardant treatment researches such as fire retardant performance, dimensional stability, and fire retardant deposit in wood observation by SEM-EDX are ongoing.

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