Effect of Using Laser Incising Treatment and Fire-Retardant Coating on *Larix kaempferi* Wood to Improve Fire Retardant Performance

Myung Sun Yang, Yeonjung Han,* and Dong Won Son

To improve fire-retardant performance of Japanese larch (Larix kaempferi) wood, this study analyzed the effect of pinholes made by laser incising and fire retardant (FR) coating on the surface of Japanese larch wood. Combustion properties such as peak heat release rate (PHRR) and total heat release (THR) of Japanese larch and Korean red pine (Pinus densiflora) wood without FRs showed similar tendencies. The comparison of the combustion properties on wood injected with an inorganic watersoluble FR under vacuum revealed that the PHRR and the THR of Korean red pine wood decreased by 37 and 62%, respectively. FR was injected into the Japanese larch specimens with pinholes on the surface and additionally coated with 5% sodium silicate and 35% potassium bromide. The results indicated a 16 to 25 and 19% reduction in PHRR and THR, respectively, compared to those without the FR. Despite the pinholes and FR coating, the FR employed in this study did not meet the standards set in Korea (THR of 8 MJ/m²). This study serves as a reference for future studies on the application of pressurized conditions and other surface treatments to improve the FR percent injection and performance of Japanese larch wood.

DOI: 10.15376/biores.17.4.6860-6874

Keywords: Fire retardant; Combustion performance; Laser incising; Surface coating; Larix kaempferi

Contact information: Forest Products and Industry Department, National Institute of Forest Science, Seoul 02455, Republic of Korea; *Corresponding author: yeonjungh@korea.kr

INTRODUCTION

Wood has long been used for various aesthetic and practical purposes in residential life as a material with excellent physical and mechanical performance (Temiz *et al.* 2008; Popescu and Pfriem 2020). As the importance of the environment has been raised along with the improvement of living standards, interest in the sustainable use of wood, which serves as a carbon sink, is increasing (Wang *et al.* 2008; U. S. Environmental Protection Agency 2020). Due to the flammable nature of wood, its use has been limited by regulations to control fire spreading (Regulation (EU) No 305 2011; KS F 2271:2021 1976; KS F ISO 5660-1:2015 2003). The combustion of polymer materials, including wood, is a complex process that requires simultaneous consideration of heat and mass transfer, fluid mechanics, and chemistry of dissolution (Dasari *et al.* 2013). Wood combustion is divided into four main stages: ignition, pyrolysis, combustion, and feedback. To control wood combustion, (1) heat shall be reduced so that combustible volatile emissions, (3) flame shall be separated from oxygen, or (4) heat flow back to wood shall be reduced to prevent further pyrolysis (Zaikov and Lomakin 1998, 2002; Bourbigot and Duquesne 2007).

Fire retardant (FR) used to protect wood from fire can reduce the flammability of wood surfaces (Kumar et al. 2015). FRs act chemically or physically on solids, liquids, and gases depending on their type and nature, and interfere with the combustion of wood at various stages such as ignition, heating, pyrolysis, and flame diffusion (Lu and Hamerton 2002; Bourbigot et al. 2004; Kiliaris and Papaspyrides 2010; Liang et al. 2013; Unlu et al. 2016). Almost all FRs for wood consist of halogens (chlorine or bromine), phosphorus, nitrogen, boric acid, borax, and inorganic metal compounds (Popescu and Pfriem 2020). Boron compounds found in the wood preservation industry are widely used as FRs for wood and wood products. Boron-based FRs provide high heat and biological resistance and have various advantages owing to their non-toxicity, easy handling, and costeffectiveness (Pedieu et al. 2012). They penetrate deeply into wood and are preserved for a long time. Among these, boric acid increases the dehydration on reacting cellulose present in the wood to reduce the amount of volatile organic compounds but has little effect on flame diffusion. The phosphorus based FRs expressed in the form of phosphate are widely used as wood coating additives for reducing inflammability and are environmentally friendly because toxic gas and smoke generation is low (Rabe et al. 2017). Sodium silicate is a viscous liquid consisting of 21 to 34% SiO₂ and 6 to 18% NaO₂ in the available silicate series known as water glass (Medina and Schledjewski 2009). Sodium silicate increases fire resistance in various materials such as paper, cement, and wood, especially in non-combustible coatings and paints (Medina and Schledjewski 2009; Lee and Thole 2018). Korean red pine (*Pinus densiflora*) and Japanese larch (*Larix kaempferi*) are the two commonly planted coniferous trees in Korea used for various purposes (Korea Forest Service 2021). Certain properties in the Japanese larch, a common building material among conifers in Korea, prevent injection of chemicals by general pressurization or depressurization treatment regardless of moisture content (Choi et al. 2011). Laser incising is being used on wood species that are difficult to inject, such as Japanese larch, to improve the penetration of chemicals (Islam et al. 2008, 2013).

In this study, as basic data for improving flame resistance of Japanese larch wood, combustion properties were measured after FR substances comprising boric acid, diammonium phosphate, ammonium borate octahydrate, sodium borate, potassium bromide, potassium carbonate, and phosphoric acid were impregnated into specimens under vacuum. To overcome the injection-resistant nature of Japanese larch, pinholes were made on the surface of the specimens using laser incasing. Finally, sodium silicate and potassium bromide were coated on the surface of Japanese larch specimens, and the difference in combustion properties was measured. The combustion properties, such as peak heat release rate (PHRR), total heat release (THR), carbon monoxide ratio to carbon dioxide (CO/CO₂), ignition time, and mass loss ratio were measured.

EXPERIMENTAL

Preparation of Specimens

Japanese larch and Korean red pine lumbers were dried according to the kilndrying schedule (T10-C4; Boone *et al.* 1988) to manufacture the specimens for measurement of combustion properties. The specimens were prepared into segments of 100 mm (longitudinal direction) \times 100 mm (radial direction) \times 10 mm (tangential direction) according to the test standard (KS F ISO 5660-1: 2015, 2003). To measure the combustion properties of the surface treatment in three iterations, three test pieces were produced for each condition. The pinholes on surface of Japanese larch specimens were made using laser incising to improve the FR percent injection. The pinholes on the surface of the specimens were spaced at 5 mm, 10 mm, or 20 mm. The specimens were conditioned at 23 °C and 50% relative humidity condition before combustion properties measurement.

Composition of Fire-Retardant Chemicals

Japanese larch and Korean red pine wood were impregnated with an inorganic water-soluble FR to improve their flame resistance. The FR was composed of distilled water as a solvent and either boric acid, diammonium phosphate, ammonium borate octahydrate, sodium borate, potassium bromide, potassium carbonate, or phosphoric acid (Son *et al.* 2014; Table 1). In order to analyze the changing in combustion properties by the surface coating of FRs, 5% sodium silicate and 35% potassium bromide were coated on the surface of Japanese larch specimens at 0.5 kg/m², each.

	Formula	CAS RN	Contribution to Composition (wt%)
Boric acid	H ₃ BO ₄	10043-35-3	0.654
Diammonium phosphate	(NH4)2HP4	7783-28-0	6.435
Ammonium borate	(NH ₄) ₂ O·5B ₂ O ₃ ·8H ₂ O	12046-03-6	1.71
octahydrate			
Sodium borate	B4Na2O7	1330-43-4	0.645
Potassium bromide	BrK	7758-02-3	6.435
Potassium carbonate	K ₂ CO ₃	584-08-7	6.435
Phosphoric acid	H ₃ PO ₄	7664-38-2	6.435
Water			Used as a solvent

Table 1. Composition of Fire-Retardant Chemicals

CAS RN: CAS Registry Number

Fire Retardant Impregnation under Vacuum

Specimens were subjected to a vacuum of -750 mmHg for 60 min to inject FRs. Before FR impregnation, a plastic net was sandwiched between the specimens to prevent them from sticking to each other. After the six specimens were positioned in the desiccator, FRs were added and then the vacuum was initiated. The FRs impregnated specimens were cured under normal pressure. After the FRs on the surface were removed, the injection amount and added amounts of the FRs were calculated.

Method for Measurement of Combustion Properties

To evaluate the fire safety of materials in case of a fire, the factors ignition point, heat release capacity, flame spread, smoke generation were determined. In Korea, the cone calorimeter test method is standardized to measure the elements of fire safety (KS F ISO 5660-1:2015, 2013). The cone calorimeter uses the principle of oxygen consumption, according to which approximately 13.1 MJ of heat is released when 1 kg of oxygen is consumed during the combustion of organic materials.

The specimens subjected to 23 °C temperature and 50% relative humidity were placed into a cone calorimeter (Fire Testing Technology Ltd., East Grinstead, UK), and radiant heat of 50 ± 0.5 kW/m² and emission flow rate of 0.024 ± 0.002 m³/s were applied to them. For specimen exposed to radiation in the horizontal direction, the heat release rate (HRR) was calculated by quantifying the concentration of oxygen measured as gases (combustion products) passing through the duct and the oxygen consumption induced by

flow rate at that time. The combustion properties PHRR, THR, CO/CO₂, and ignition time were also measured.

HRR is defined as instantaneous heat generated per surface area where a material burns and can be calculated using the difference between measured oxygen concentration and atmospheric oxygen concentration (Eq. 1),

$$\dot{q}(t) = 1.10 \times \left(\frac{\Delta h_c}{r_0}\right) \times C \sqrt{\frac{\Delta P}{T_e}} \times \frac{X_{O_2}^0 - X_{O_2}}{1.105 - 1.5X_{O_2}}$$
(1)

where \dot{q} is the heat release rate (kW), Δh_c is the net heat of combustion (kJ/g), r_0 is the stoichiometric oxygen–fuel mass ratio, *C* is the orifice flow meter calibration constant (m^{1/2}·g^{1/2}·K^{1/2}), ΔP is the orifice meter pressure differential (Pa), T_e is the absolute temperature of gas at the orifice meter (K), $X_{o_2}^0$ is the initial value of oxygen analyzer reading, and X_{o_2} is oxygen analyzer reading (mole fraction of oxygen).

If the value of $\Delta h_c/r_0$ is not known in Eq. 1, it can be assumed to be 13.1 MJ/kg. Moreover, if $X_{o_2}^0$ is the average oxygen analyzer value obtained by the reference measurement for 1 min, the HRR per unit area can be corrected as shown in Eq. 2,

$$\dot{q}_A(t) = \frac{\dot{q}(t)}{A_s} \tag{2}$$

where A_s is the initially exposed surface area of the specimens and is equal to 0.0088 m², \dot{q}_A is the heat release rate per unit area (kW/m²), and *t* is time (s).

A THR value represents the sum of the heat released as the combustion progresses after ignition on the surface of the material and is calculated as the sum of the HRR expressed as a function of time to the surface area of the specimen (Eq. 3),

$$Q_{A,total} = \sum_{1}^{t} \dot{q}_A , \qquad (3)$$

where $Q_{A, \text{total}}$ is the total heat released per unit area during the entire test (MJ/m²) and *t* is time the duration of the entire test.

In order to compare relative hazards of materials showing different combustion rates, CO/CO₂, a factor for comparing the amount of CO produced when the same amount of CO₂ is generated, was measured. The ignition time is defined as the time when the flame begins to occur on the surface of the material, and it is an element that can evaluate the combustibility and the possibility of flame diffusion along with the HRR. Ignition time can be calculated as shown in Eq. 4,

$$t_{ig} = C \cdot k \cdot \rho \cdot c \left(\frac{T_{ig} - T_s}{q^{"}} \right)^2 \tag{4}$$

where t_{ig} is ignition time (s), *C* is constant, *k* is thermal conductivity (kW·m⁻¹·°C⁻¹), T_{ig} is temperature ignition (°C), T_s is ambient temperature (°C), and *q*'' is the radiation intensity (heat flux) (kW/m²).

Method for Determining Toxicity Index

Nine types of gases were measured using a direct-reading individual gas detection tube for completely combusted specimens. The concentration of the gas detected by combusting 1 g of specimen was converted to 100 g of substance, and the toxicity index was calculated by utilizing the lethal concentration (toxic concentration fatal to human at a 30 min exposure time) of nine types of gases according to the Naval Engineering Standard (NES 713, 1985; Eq. 5),

$$TI = \frac{C_{g1}}{C_{f1}} + \frac{C_{g2}}{C_{f2}} + \dots + \frac{C_{gn}}{C_{fn}},$$
(5)

where $C_g = C \times 100 \times V/m$, *C* is the correction concentration of gas in the chamber (ppm), *V* is the volume of test chamber (m³), *m* is the fire test weight (g), *C_f* is the concentration of the gas considered fatal to man during a 30 min exposure time (ppm), and 1, 2, 3... *n* represents each of the gasses detected.

RESULTS AND DISCUSSION

Combustion Properties Japanese Larch and Korean Red Pine Wood

Combustion properties, such as PHRR, THR, CO/CO₂, total oxygen consumed (TOC), ignition time, and weight loss rates, of Japanese larch and Korean red pine wood are presented in Table 2. The results of the PHRR and THR are shown in Fig. 1. In both species, the HRR increased rapidly at the start of pyrolysis at the initial stage of combustion and then gradually decreased, reaching a second peak in the process of secondary pyrolysis (Fig. 1(a)). After reaching the PHRR, the HRR decreased continuously as the combustion of the specimen gradually decreased owing to the adiabatic effect of the carbonized layer formed during combustion (Pearce et al. 1981). The time at which the PHRR occurred as an element of combustion inhibition was measured at 520 and 298 s in Japanese larch and Korean red pine wood, respectively. The THR of Japanese larch was higher than that of Korean red pine (Fig. 1(b)). Generally, since the specific gravity of Japanese larch (0.45 to (0.50) was higher than that of Korean red pine (0.40 to 0.45), the amount of wood combusted at the same volume and the oxygen demand were higher (Chong and Park 2008). This trend was similar to the results of a combustion performance test on wood-containing insulation materials, which showed a correlation between the THR and density of the materials (Lee and Kim 2003).

	Japanese Larch Wood	Korean Red Pine Wood
PHRR (kW/m ²)	180.3	192.1
THR (MJ/m ²)	56.7	40.2
Mean CO yield	0.0124	0.0067
Mean CO ₂ yield	0.950	0.838
CO/CO ₂ (%)	1.305	0.800
TOC (g)	43.0	25.5
Ignition time (s)	15.0	19.4
Initial weight (g)	59.9	39.9
Weight lost (g)	42.2	33
Weight loss rate (%)	70.5	82.7

Table 2. Combustion Properties of Japanese Larch Wood and Korean Red Pine

 Wood

PHRR: Peak heat release rate; THR: Total heat release; TOC: Total oxygen consumed

The performance level of FR materials in the Korean industrial standard (KS F ISO 5660-1:2015, 2013) is defined as "PHRR not exceeding 200 kW/m² for more than 10 s for 5 min and THR being less than 8 MJ/m² for 5 min." In the combustion properties of Japanese larch and Korean red pine wood, the PHRR did not exceed 200 kW/m² for more than 10 s in a row, but the THR was greater than the 8 MJ/m² standard and failed to pass the performance level of FR materials.



Fig. 1. Changes in the heat release rate and total heat release of Japanese larch wood and Korean red pine during combustion: (a) Heat release rate and (b) Total heat release.

The ratio of CO/CO₂ generation generally indicates the combustion aspect of materials (Hull and Paul 2007). CO₂ is a natural product during combustion, whereas CO is generated because the contact between the burning material and oxygen in the air is restricted by the carbonized layer generated on the wood surface during combustion (Byrne and Nagle 1997; Seo *et al.* 2017). Unlike polymeric materials such as synthetic resins, wood hardly produces harmful gases; therefore, the toxicity of the combustion gas is determined by the release of CO accompanied by CO₂. The average CO yield when Japanese larch and Korean red pine wood were exposed to radiant heat for 30 min was 0.0124 and 0.0067, respectively. Smoke generated during the combustion of Korean red pine wood was less harmful than that generated during Japanese larch wood combustion.

The ignition time, which is a factor indicating the ignition characteristics of materials during combustion, was 15.0 sand 19.4 s for Japanese larch and Korean red pine wood, respectively.

Changes in Combustion Properties by Impregnation of Fire Retardant

FR manufactured in the ratio presented in Table 1 was impregnated into Japanese larch and Korean red pine specimens under vacuum of -750 mmHg, and the results of the combustion properties are presented in Table 3. The combustion properties of lignocellulosic materials were affected by lignin content. The materials with higher lignin content exhibit a lower HRR (Kozłowski and Władyka-Przybylak 2008). Thus, a high content of cellulose could increase the flammability of the lignocellulosic materials (Dorez et al. 2014). The lignin content of Japanese larch and Korean red pine vary depending on the growth area and the sampling time, but their values were within very similar ranges, 23.8 to 28.2% in Japanese larch and 24.2 to 26.8% in Korean red pine wood (Park et al. 2017). Accordingly, the flame resistance of the Japanese larch wood and the Korean red pine wood differed depending on the FR injection rate. The absorption and injection rate of FR were 5.71 kg/m³ and 5.95% for Japanese larch and 25.5 kg/m³ and 28.9% for Korean red pine, respectively. Due to the high injection rate of Korean red pine wood under vacuum, the PHRR decreased by approximately 37% from 192.1 to 120.8 kW/m², and the THR decreased by approximately 62% from 40.2 to 15.2 MJ/m². Moreover, since the injection rate of the Japanese larch wood is relatively low under vacuum, no substantial differences were observed in the combustion properties compared with the case where the FR was not injected.

Pits have a major influence on softwood permeability (Siau 1995). Flow across pits can be impeded by aspiration by deposition of extractives on the membrane (Bao *et al.* 2001; Islam *et al.* 2009). FR injection also significantly affects pit condition by inducing aspirations that block the fluid flow. The Japanese larch has a high proportion of heartwood with a high extractive content, rendering FR injection difficult compared with other tree species.

	Japanese Larch Wood	Korean Red Pine Wood
PHRR (kW/m ²)	180.8	120.8
THR (MJ/m ²)	59.5	15.2
Mean CO yield	0.0318	0.0414
Mean CO ₂ yield	1.16	0.838
CO/CO ₂ (%)	2.74	4.94
TOC (g)	38.7	25.3
Ignition time (s)	27.0	25.0
Initial weight (g)	64.2	46.8
Weight lost (g)	48.2	31.2
Weight loss rate (%)	75.1	66.7
FR absorption (kg/m ³)	5.71	25.5
FR percent injection (%)	5.95	28.9

Table 3. Combustion Properties of Japanese Larch Wood and Korean Red Pine

 Wood Impregnated with Fire-Retardant

PHRR: Peak heat release rate; THR: Total heat release; TOC: Total oxygen consumed; FR: Fire resistance

Difference in Percent Injection by Laser Incising Treatment on the Surface of Japanese Larch Wood

Pinholes were added and spaced at 5, 10, and 20 mm on the surface of the specimens to improve the percent injection of the Japanese larch wood (Fig. 2). The number of pinholes generated on the surface ranged from 25 (2,500 ea./m²) to 400 (40,000 ea./m²) depending on the spacings, and the size of pinholes was 0.5 mm. A change in the FR percent injection of Japanese larch specimens by the spacings of pinholes through laser incising treatment is presented in Table 4.



Fig. 2. Pinhole formation of the surface of Japanese larch specimens by laser incising treatment

The percentages of injection of FR at the three pinhole spacings on the surface of the specimen were 20.9% (5 mm), 10.4% (10 mm), and 8.2% (20 mm). Although FR percent injection of the Japanese larch wood increased under vacuum compared to wood surface without pinholes, it showed a lower value than the 28.9% percent injection of Korean red pine wood without pinholes. Generally, pinholes on the surface can impact the strength characteristics of the wood, and the surface processing with pinholes less than 5 mm spacing may negatively affect the appearance of the FR products.

Table 4. Difference in the Impregnation Ra	te by Pinhole Formation of the
Surface of Japanese Larch Specimens	

Distance between holes (mm)	5	10	20
Number of holes	400	81	25
Hole density on surface (ea./m ²)	40,000	8,100	2,500
Percent injection (%)	20.9	10.4	8.2

Change in Combustion Properties by Fire-Retardant Coating on the Surface of Japanese Larch Wood

FR was injected into the Japanese larch specimens with pinholes spaced at 5 mm. In addition, two types of FR chemicals, 5% sodium silicate and 35% potassium bromide, were coated on the surface to measure the change in combustion properties (Table 5). The FR chemicals were applied at 500 g/m² each by brushing until the wood surface was sufficiently soaked, and the surface was allowed to dry for 24h at 23°C and a 50% relative humidity; this process was repeated twice.

The PHRR and THR between Japanese larch wood specimens with and without FR were compared (Fig. 3). Compared with the specimens without FR, the specimens with two types of FR chemicals showed delayed pyrolysis in the early stages of combustion. FR

coated on surface of specimens affects the degradation pathway of the cellulose and leads to charring and incomplete combustion of specimens, limiting their contribution to the heat evolved during combustion (Dorez *et al.* 2014). After reaching a sufficiently high temperature (300 s), the specimens with FR were stacked with increasing number of char layers to limit combustion. Consequently, the PHRR of the specimen with FR appeared low (Fig. 3(a)). The PHRRs in the specimens coated with 5% sodium silicate and 35% potassium bromide were 149.9 and 134.9 kW/m², respectively. The THR was 46.2 MJ/m² for both types of FR chemicals. The combustion properties between the two types of FR chemicals used for surface coating did not show major differences. Compared to Japanese larch wood without FR, specimen with FR showed a decrease in PHRR and THR by approximately 20%, which indicates an improvement in FR performance. However, the THR value in this study was beyond the standard of 8 MJ/m² set for FR materials in Korea.

Table 5. Combustion Properties of Japanese Larch Wood Surface-Coated with

 Chemicals

	Japanese Larch Specimens Impregnated with Fire Retardant After Laser Incising Treatment on the Surface		
	5% Sodium silicate coating	35% Potassium bromide coating	
PHRR (kW/m ²)	149.9	134.9	
THR (MJ/m ²)	46.2	46.2	
Mean CO yield	0.0513	0.025	
Mean CO ₂ yield	1.09	0.90	
CO/CO ₂ (%)	4.71	2.78	
TOC (g)	30.3	28.6	
Ignition time (s)	32.0	39.0	
Initial weight (g)	63.3	63.6	
Weight lost (g)	30.3	43.4	
Weight loss rate (%)	47.87	68.24	
FR absorption (kg/m ³)	19.3	18.9	
FR percent injection (%)	20.9	20.8	

PHRR: Peak heat release rate; THR: Total heat release; TOC: Total oxygen consumed; FR: Fire resistance



Fig. 3. Changes in the heat release rate and total heat release during combustion of Japanese larch wood surface-coated with chemical: (a) Heat release rate and (b) Total heat release (PHRR: Peak heat release rate)

When exposed to heat, organic materials generate various types of fire gases and smoke (Biswas et al. 2007). The major toxicant is carbon monoxide (CO), which is accompanied by carbon dioxide (CO₂). CO is produced by incomplete combustion of organic material at low temperature in early stages of fire. As the fire intensifies, CO₂ is formed under high temperature conditions, which depends on the availability of oxygen in the environment. The amount of CO₂ emitted during combustion of organic materials tends to be similar to that of the THR (Chuang et al. 2013). Previous studies have shown that CO₂ emissions decrease when wood products are coated with FR (Almeras et al. 2003; Chuang et al. 2013). However, the results in Table 5 confirm that the CO/CO₂ results were higher in specimens injected with FR than in those without FR. The toxicity index of combustion gases was analyzed in accordance with the UK Naval Engineering Standard 713 (NES 713, 1985), to experiment with the toxicity of smoke generated during wood combustion (Table 6). The amount of CO_2 measured using the detection tube was in the range of 650 to 750 ppm in three types of specimens, and trace amounts of NO₂ were detected. The toxicity index was 0.183, 0.251, and 0.200 for control, sodium silicate surface coating, and potassium bromide surface coating, respectively. These values were very low as compared to other toxicity indices for building material such as glued laminated timber (1.77), medium density fiberboard (5.85), and plywood (5.30) (Kim and Lee 2016) and insulation materials such as polyethylene (18.24), polyurethane (12.35), and fiberglass (6.95) (Liang and Ho 2007).

Table 6. Concentration (ppm) of Gases and Naval Engineering Standard 713Toxicity Index

Division (ppm)	Toxic Concentration Fatal to Human at 30 min Exposure Time according to NES-713	Control (Japanese Larch Wood)	5% Sodium Silicate Coating	35% Potassium Bromide Coating
Toxicity index		0.183	0.251	0.200
CO ₂ (ppm)	10,000	650	750	750
CO (ppm)	4,000	0	0	0
NO + NO ₂ (ppm)	250	0.2	0.2	0.2
C ₆ H₅OH (ppm)	250	0	0	0
SO ₂ (ppm)	400	0	0	0
NH₃ (ppm)	550	0	0	0
HCHO (ppm)	500	0	0	0
HCN (ppm)	150	0	0	0

Through this study, it was confirmed that vacuum is an unsuitable method for injecting FR into the Japanese larch wood. Additional studies utilizing surface treatments and pressurized conditions are required to improve the FR percent injection and FR performance of Japanese larch wood.

CONCLUSIONS

- 1. Combustion properties such as peak heat release rate (PHRR), total heat release (THR), CO/CO₂ ratio, total oxygen consumed (TOC), ignition time, and weight loss rates of Japanese larch and Korean red pine wood were measured. The PHRR of Japanese larch and Korean red pine wood without FR were 180.3 and 192.1 KW/m², and the THR was 56.7 MJ/m² and 40.2 MJ/m², respectively. The combustion properties of two species showed similar trends without much difference.
- 2. The percentages of injection of Japanese larch and Korean red pine wood after the impregnation of water-soluble FR into the specimens under vacuum of -750 mmHg were 5.95 and 28.9%, respectively. A 37 and 62% decrease in the PHRR and THR, respectively, was observed for Korean red pine wood upon FR injection, whereas the values of Japanese larch wood hardly changed after the FR injection.
- 3. The percentages of injection of FR at the three pinhole densities on the surface of the Japanese larch specimen were 20.9% (5 mm), 10.4% (10 mm), and 8.2% (20 mm). The PHRR and THR of Japanese larch specimen impregnated with FR after the laser incising treatment and additionally coated with 5% sodium silicate and 35% potassium bromide were reduced by 16 to 25 and 19%, respectively, compared with those without the FR impregnation.
- 4. To assess fire resistance of Japanese larch wood, this study analyzed the effect of pinholes made by laser incising and FR coating on the wood surface. Despite the presence of pinholes and FR coating, the wood did not meet the standards set for FR materials in Korea (THR of 8 MJ/m²).

ACKNOWLEDGMENTS

This work was supported by the National Institute of Forest Science (NIFoS) grant funded by the Korea government.

REFERENCES CITED

- Almeras, X., Le Bras, M., Hornsby, P., Bourbigot, S., Marosi, G. Y., Keszei, S., and Poutch, F. (2003). "Effects of fillers on the fire retardancy of intumescent polypropylene compounds," *Polym. Degrad. Stab.* 82(2), 325-331. DOI: 10.1016/S0141-3910(03)00187-3
- Bao, F., Lu, J., and Zhao, Y. (2001). "Effect of bordered pit torus position on permeability in Chinese Yezo spruce," *Wood Fiber Sci.* 33(2), 193-199. DOI: wfs.swst.org/index.php/wfs/article/view/1090
- Biswas, B., Kandola, B. K., Horrocks, A. R., and Price, D. (2007). "A quantitative study of carbon monoxide and carbon dioxide evolution during thermal degradation of flame retarded epoxy resins," *Polym. Degrad. Stab.* 92(5), 765-776. DOI: 10.1016/j.polymdegradstab.2007.02.006
- Boone, R. S., Kozlik, C. J., Bois, P. J., and Wengert, E. M. (1988). Dry Kiln Schedules for Commercial Woods: Temperate and Tropical (General Technical Report FPL-GTR-57), Madison, WI, USA. DOI: 10.2737/FPL-GTR-57
- Bourbigot, S., Le Bras, M., Duquesne, S., and Rochery, M. (2004). "Recent advances for intumescent polymers," *Macromol. Mater. Eng.* 289(6), 499-511. DOI: 10.1002/mame.200400007
- Bourbigot, S., and Duquesne, S. (2007). "Fire retardant polymers: Recent developments and opportunities," *J. Mater. Chem.* 17(22), 2283-2300. DOI: 10.1039/B702511D
- Byrne, C. E., and Nagle, D. C. (1997). "Carbonization of wood for advanced materials applications," *Carbon* 35(2), 259-266. DOI: 10.1016/S0008-6223(96)00136-4
- Choi, Y. S., Oh, S. M., and Kim, G. H. (2011). "Evaluation of pretreatment moisture content and fixation characteristics of treated wood for pressure treatment of Japanese red pine and Japanese larch skin timber with ACQ, CUAZ and CuHDO," J. Korean Wood Sci. Technol. 39(6), 481-489. DOI: 10.5658/WOOD.2011.3936.481
- Chong, S. H., and Park, B. S. (2008). *Wood Properties of the Useful Tree Species Grown in Korea*, National Institute of Forest Science, Seoul, Korea.
- Chuang, C. S., Yang, T. H., Tsai, K. C., Tseng, T. Y., and Wang, M. K. (2013). "Fire retardancy and CO/CO₂ emission of intumescent coatings on thin plywood panel with waterborne vinyl acetate-acrylic resin," *Wood Sci. Technol.* 47(2), 353-367. DOI: 10.1007/s00226-012-0491-x
- Dasari, A., Yu, Z. Z., Cai, G. P., and Mai, Y. W. (2013). "Recent developments in the fire retardancy of polymeric materials," *Prog. Polym. Sci.* 38(9), 1357-1387. DOI: 10.1016/j.progpolymsci.2013.06.006
- Dorez, G., Ferry, L., Sonnier, R., Taguet, A., and Lopez-Cuesta, J. M. (2014). "Effect of cellulose, hemicellulose and lignin contents on pyrolysis and combustion of natural fibers," *J. Anal. Appl. Pyrol.* 107, 323-331. DOI: 10.1016/j.jaap.2014.03.017
- Hull, T. R., and Paul, K. T. (2007). "Bench-scale assessment of combustion toxicity-A critical analysis of current protocols," *Fire Saf. J.* 42(5), 340-365. DOI: 10.1016/j.firesaf.2006.12.006

- Islam, M. N., Ando, K., Yamauchi, H., and Hattori, N. (2008). "Comparative study between full cell and passive impregnation method of wood preservation for laser incised Douglas fir lumber," *Wood Sci. Technol.* 42(4), 343-350. DOI: 10.1007/s00226-007-0168-z
- Islam, M. N., Ando, K., Yamauch, H., and Hattori, N. (2009). "Effects of species and moisture content on penetration of liquid in laser incised lumber by the passive impregnation method," *Eur. J. Wood Prod.* 67(2), 129-133. DOI: 10.1007/s00107-008-0292-y
- Islam, M. N., Ando, K., Yamauchi, H., and Hattori, N. (2013). "Impregnation of preservative and fire retardants into Japanese cedar lumber by passive impregnation," *BioResources* 8(1), 395-404. DOI: 10.15376/biores.8.1.395-404
- Kiliaris, P., and Papaspyrides, C. D. (2010). "Polymer/layered silicate (clay) nanocomposites: an overview of flame retardancy," *Progr. Polym. Sci.* 35(7), 902-958. DOI: 10.1016/j.progpolymsci.2010.03.001
- Kim, J. B., and Lee, S. Y. (2016). "Toxicity evaluation of the combustion products from synthetic wood as internal finish," *Fire Sci. Eng.* 30(2), 7-18. DOI: 10.7731/KIFSE.2016.30.2.007
- Korea Forest Service (2021). *Statistical Yearbook of Forestry 2021*. Korea Forest Service, Daejeon, Korea.
- Kozłowski, R., and Władyka-Przybylak, M. (2008). "Flammability and fire resistance of composites reinforced by natural fibers," *Polym. Adv. Technol.* 19(6), 446-453. DOI: 10.1002/pat.1135
- KS F 2271: 2021 (1976). "Testing method for gas toxicity of finish materials of buildings," Korean Standards Association, Seoul, Korea.
- KS F ISO 5660-1:2015 (2003). "Reaction to fire test Heat release, smoke production and mass loss rate – Part 1. Heat release rate (Cone calorimeter method) and smoke production rate (dynamic measurement)," Korean Standards Association, Seoul, Korea.
- Kumar, S. P., Takamori, S., Araki, H., and Kuroda, S. (2015). "Flame retardancy of claysodium silicate composite coatings on wood for construction purposes," *RSC Adv*. 5(43), 34109-34116. DOI: 10.1039/C5RA04682C
- Lee, K. W., and Kim, K. E. (2003). "Fire characteristics of plastic insulating materials from cone calorimeter test," *Fire Sci. Eng.* 17(1), 76-83.
- Lee, S. J., and Thole, V. (2018). "Investigation of modified water glass as adhesive for wood and particleboard: Mechanical, thermal and flame-retardant properties," *Eur. J. Wood Prod.* 76(5), 1427-1434. DOI: 10.1007/s00107-018-1324-x
- Liang, H. H., and Ho, M. C. (2007). "Toxicity characteristics of commercially manufactured insulation materials for building applications in Taiwan," *Constr. Build. Mater.* 216(6), 1254-1261. DOI: 10.1016/j.conbuildmat.2006.05.051
- Liang, S., Neisius, N. M., and Gaan, S. (2013). "Recent developments in flame retardant polymeric coatings," *Prog. Org. Coat.* 76(11), 1642-1665. DOI: 10.1016/j.porgcoat.2013.07.014
- Lu, S. Y., and Hamerton, I. (2002). "Recent developments in the chemistry of halogenfree flame retardant polymers," *Progr. Polym. Sci.* 27(8), 1661-1712. DOI: 10.1016/S0079-6700(02)00018-7
- Medina, L. A., and Schledjewski, R. (2009). "Water glass as hydrophobic and flame retardant additive for natural fiber reinforced composites," *J. Nanostruct. Polym. Nanocompos.* 5, 107-114.

NES 713 (1985). "Determination of the toxicity index of the products of combustion from small specimens of materials, Naval Engineering Standard 713," Issue 3, Ship Department, Foxhill, Bath, UK.

Park, S. Y., Kim, J. C., Kim, J. H., Yang, S. Y., Kwon, O., Yeo, H., Cho, K. C., and Choi, I. G. (2017). "Possibility of wood classification in Korean softwood species using near-infrared spectroscopy based on their chemical compositions," *J. Korean Wood Sci. Technol.* 45(2), 202-212. DOI: 10.5658/WOOD.2017.45.2.202

Pearce, F. M., Khanna, Y. P., and Raucher, D. (1981). "Chap. 8 – Thermal analysis in polymer flammability," in: Turi, E. A. (ed.), *Thermal Characterization of Polymeric Materials*, Academic Press, New York, pp. 793-843.

Pedieu, R., Kouba, A., Riedl, B., Wang, X. M., and Deng, J. (2012). "Fire-retardant properties of wood particleboards treated with boric acid," *Eur. J. Wood Prod.* 70(1-3), 191-197. DOI: 10.1007/s00107-011-0538-y

Popescu, C. M., and Pfriem, A. (2020). "Treatments and modification to improve the reaction to fire of wood and wood-based products – An overview," *Fire Mater.* 44(1), 100-111. DOI: 10.1002/fam.2779

Regulation (EU) No 305/2011 of the European Parliament and of the Council of 9 March 2011 laying down harmonised conditions for the marketing of construction products and repealing Council Directive 89/106/EEC. (2011). Official Journal of the European Union (https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32011R0305&rid=2).

Seo, H. J., Hwang, W., and Lee, M. C. (2017). "Fire properties of *Pinus densiflora* utilizing fire-retardant chemicals based on borated and phosphorus (I) – Combustion characteristics," *BioResources* 12(3), 5417-5427. DOI: 10.15376/biores.12.3.5417-5427

- Siau, J. F. (1995). *Wood: Influence of Moisture on Physical Properties*, Dept. of Wood Sci. and Forest Prod, Virginia Polytechnic Institute and State Univ., Blacksburg, VA.
- Son, D. W., Park, S. B., Hwang, W. J., and Kang, M. R. (2014). "Composition for wood fire retardants, flame retardant and method for manufacturing thereof," Korea Patent 10-1597986-0000, filed April 28, 2014, issued February 22, 2016.
- Temiz, A., Gezer, E. D., Yildiz U. C., and Yildiz, S. (2008). "Combustion properties of alder (*Alnus glutinosa* L.) Gaertn. subsp. *barbata* (C.A. Mey) Yalt. and southern pine (*Pinus sylvestris* L.) wood treated with boron compounds," *Construct. Build. Mater*. 22(11), 2165-2169. DOI: 10.1016/j.conbuildmat.2007.08.011
- Unlu, S. M., Tayfun, U., Yildirim, B., and Dogan, M. (2016). "Effect of boron compounds on fire protection properties of epoxy based intumescent coating," *Fire Mater.* 41(1), 17-28. DOI: 10.1002/fam.2360
- U.S. Environmental Protection Agency (2020). Documentation for Greenhouse Gas Emission and Energy Factors Used in the Waste Reduction Model (WARM), U. S. Environmental Protection Agency, Office of Resource Conservation and Recovery, (https://www.epa.gov/sites/default/files/2020-12/documents/warm background v15 10-29-2020.pdf).

Wang, F., Zhang, Z., Wang, Q., and Tang, J. (2008). "Fire-retardant and smoke suppressant performance of an intumescent waterborne amino resin fire-retardant coating for wood," *Front. For. China* 3(4), 487-492.

DOI: 10.1007/s11461-008-0075-y

- Zaikov, G. E., and Lomakin, S. M. (1998). "Polymer flame retardancy: A new approach," J. Appl. Polym. Sci. 68(5), 715-725. DOI: 10.1002/(SICI)1097-4628(19980502)68:5<715::AID-APP4>3.0.CO;2-R
- Zaikov, G. E., and Lomakin, S. M. (2002). "Ecological issue of polymer flame retardancy," J. Appl. Polym. Sci. 86(10), 2449-2462. DOI: 10.1002/app.10946

Article submitted: August 22, 2022; Peer review completed: Sept. 18, 2022; Revised version received and accepted: October 12, 2022; Published: October 20, 2022. DOI: 10.15376/biores.17.4.6860-6874