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Effect of grooving pretreatment on flame retardant vacuum-pressure impregnation performance of full-size timbers

Eun-Suk Jang[1,3], Seok-Un Jo[2] and Hee-Jun Park[2*]

Abstract

We reported in a previous study that wood fiber orthogonal grooving improves flame retardant vacuum-pressure impregnation performance. However, it was limited to small sample sizes. We extended our previous study to investigate the effect of grooving on flame retardant impregnation in Japanese cedar (*Cryptomeria japonica* D. Don.) and Korean larch (*Larix kaempferi* (Lamb.) Carriere) full-size timber [2,400 mm(L) × 100 mm(W) × 22 mm(T)], which are widely used as a building interior lining. Japanese cedar had higher open-pore porosity and gas permeability than Korean larch. As a result, Japanese cedar had no significant difficulty impregnating the flame retardant, even without a grooving pretreatment process. The flame retardant's impregnating performance in Korean larch control was insignificant, but the impregnating ability was improved after fiber orthogonal grooving. In conclusion, Japanese cedar can meet the fire safety standards announced by the Ministry of Land, Infrastructure and Transport without groove processing before flame retardant impregnation. On the other hand, Korean larch had to be impregnated with a flame retardant after grooving processing in the orthogonal direction of the fiber to achieve fire safety performance. As a result, this grooving pretreatment is a simple and practical technique that can be used immediately on impervious commercial wood.

Keywords Vacuum-pressure impregnation, Korean larch, Japanese cedar, Flame retardant, Grooving, Building interior lining

Introduction

Forests play a vital role in mitigating climate change by serving as significant carbon sinks. Through the process of photosynthesis, trees absorb carbon dioxide (CO_2) from the atmosphere and convert it into organic matter, effectively sequestering and storing carbon [1]. Even if trees are cut down and used as wood, carbon dioxide is not separated. Therefore, The use of woody materials has the effect of increasing the carbon sink in the atmosphere, which is sustainable forestry [2, 3].

Human use of wood begins in prehistoric times [4]. Wood is used in various products, including furniture, sporting goods, and kitchen utensils, but the most common use is in the construction sector [5, 6]. Although various materials have been developed in construction, wood is still a natural and sustainable material widely used as a building material [7].

Wood comprises cellulose, hemicellulose, and lignin [8–10]. These are organic materials and are combustible. This is one of the fatal disadvantages of wood as a building material. Thus, flame retardant treatment for wood products is essential to ensure the fire safety of buildings [11].

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Flame retardants applied to wood contain sulfates, ammonium phosphates, chlorides, oxides, borates, halogens, etc. To protect it from fire, these are laminated, coated, or impregnated into wood [12].

Fire retardant coatings are a simple, cost-effective way to safeguard wood against fire damage [13, 14]. When exposed to fire, these coatings undergo a transformation, expanding and forming a protective layer of carbonized bubbles. This carbonized layer acts as a barrier, effectively shielding the wood from heat and oxygen and delaying the onset of wood combustion[15]. However, it is important to note that the primary drawback of this method is that the flame retardant properties are compromised if the coating layer is damaged or if the carbonized layer detaches during a fire incident [12].

The vacuum pressure impregnation process is a flame-retardant treatment technique that involves injecting a flame-retardant solution into wood under varying pressure conditions. In comparison to coating treatment, it offers enhanced fire protection as it allows for the application of fire retardant to the interior of the wood [12, 16, 17].

The performance of the vacuum pressure impregnation process depends on the permeability of the wood, the chemical properties of the flame retardant, the impregnation pressure and time [18]. The permeability of wood is influenced by its pore structure, particularly the level of "through-pore porosity" [19–21]. Woods with high through-pore porosity exhibit good permeability, enabling effective impregnation [22]. Furthermore, sapwood is more readily impregnated compared to heartwood. The reduced impregnation ability of heartwood can be attributed to bordered pit aspiration during heartwood formation [23]. Bordered pit aspiration makes increases the closed pore porosity, creating obstacles for the impregnation of flame retardants [24].

To address the impermeability of wood, variable physicochemical treatment methods such as thermal [25, 26], ultrasonic [27, 28], microwave [29, 30], oxidation [31] and steam explosion [32] have been introduced. These pretreatments can improve the flame retardant's impregnation performance by increasing the wood pores' fluidity. However, there is a financial burden of preparing a separate facility for such processing.

Boring and kerfing are widely used as pretreatment methods that can better impregnate wood with flame retardants [33, 34]. In addition, the process of drilling tiny holes in the wood surface using a laser was introduced. This is a way to improve the containment performance of flame retardants by increasing the porosity of the wood surface without significantly damaging the wood surface texture [35, 36].

The simpler the pretreatment method, the more time and budget savings in the manufacturing process. Therefore, our research group explored how to expect the maximum impregnation effect using a relatively easy pretreatment method. In prior research, we established that the longitudinal and transverse grooving process, employed as a pretreatment technique in vacuum pressure impregnation, enhanced the flame-retardant impregnation efficacy specifically in larch wood. However, the scope of this previous study was limited to a small sample size [37]. To build upon these findings, we expanded the scope of our research to include full-size timber measuring 2400 mm (L) × 100 mm (W) × 22 mm (T), which is widely utilized as a material in building construction. We assessed the impact of the grooving process on the flame-retardant impregnation performance of this full-sized timber.

Materials and methods

Sample preparation

This study was air-dried Korean larch (*Larix kaempferi* (Lamb.) Carriere) and Japanese cedar (*Cryptomeria japonica* D. Don.) supplied from Yuseong timber (Iksan, Korea). Their size was 2400 mm (L) × 100 mm (W) × 22 mm (T), and the average moisture content was 12.91%. The bulk density of Korean larch was 0.54 g/cm^3, and that of Japanese cedar was 0.33 g/cm^3.

Porosity and gas permeability

The impregnation of flame retardants depends on the pore structure and permeability of wood species [38, 39]. Therefore, we investigated the closed-pore porosity (ϕ_{closed}) and open-pore porosity (ϕ_{open}) and gas permeability of cedar and larch. The total porosity (ϕ_{total}) of wood can be obtained by the relationship between the true density and bulk density of wood (Eq. 1) [27]:

$$\phi_{total} = \left(1 - \frac{\rho_b}{\rho_t}\right) \times 100 \quad (1)$$

where ρ_b is the density of specimen and ρ_t is the true density of wood (wood's true density is assumed to be 1.5 g/cm^3 [40].

Open-pore volume can be measured using a gas pycnometer (PYC-100A-1, PMI, USA). A gas pycnometer consists of a sample cell and a reference cell. A valve connects the two cells. A wood sample is put into the sample cell and the valve is closed. The reference cell is filled with helium gas at a pressure of about 30 psi (0.2 MPa). When the pressure is stable, open the valve. Helium from the reference cell moves to the sample cell and penetrates into the open cell of the wood, causing a pressure drop. From this pressure change, the open pore volume of the

wood sample is calculated using Boyle's Law. From this value, closed-pore porosity and open-pore porosity can be obtained (Eqs. 2 and 3) [21]:

$$\phi_{\text{closed}} = \left(\frac{{\rho_{\text{p}}}^{-1} - {\rho_{\text{t}}}^{-1}}{{\rho_{\text{b}}}^{-1} - {\rho_{\text{t}}}^{-1}} \right) \times 100 \quad (2)$$

where ρ_{p} is the density of specimen using a gas pycnometer (g/cm^3):

$$\phi_{\text{open}} = \phi_{\text{total}} - \phi_{\text{closed}} \quad (3)$$

The capillary flow porometer (model: CFP-1200AEL; PMI, USA) was employed to measure the gas permeability. The flow rate was measured while applying a pressure of 15 psi (0.1 MPa) in the vertical direction of the sample. The Darcy permeability constant of the specimens was calculated using the following equation [23]:

$$D = 8FTV/\pi d^2(P^2 - 1) \quad (4)$$

where D is the Darcy permeability constant, F is the flow (cm^3/s), T is the sample thickness (cm), V is the viscosity of air (cp), d is the sample diameter (cm), and P is the pressure (psi).

Grooving process

Figure 1 shows a picture of the wood for the grooving model used in this study. Groove processing was performed with a width of 6 mm and depth of 7 mm, and there were consist of four models. M1 is unprocessed (control), M2 is grooved in the fiber direction (at 30 mm intervals), M3 was grooved at a fiber orthogonal direction (100 mm intervals), and M4 was mixed grooving of M2 + M3.

Flame retardant

In this study, a water-soluble flame retardant supplied by Safe Wood Co., Ltd. (Jeonju, Korea) was used. Its constituents were 12% dibasic ammonium phosphate, 8% ammonium polyphosphate, 7% anhydrous sodium borate and 3% other ingredients. The flame retardant was formulated with a resin content of approximately 30%. Its specific gravity of 1.16 at 25 °C, and a pH of 8.3. This study used a 3% dilution of water-soluble blue ink to facilitate a visual assessment of the flame retardant's impregnation.

Vacuum pressure impregnation process

The impregnation process in this study utilized the same wood vacuum pressure impregnation machine as in our previous study (Fig. 2). The yellow circles indicate samples located inside the chamber. The wood with each grooving type was placed in a chamber and decompressed at − 0.1 MPa for about 5 min using a vacuum pump. After that, the flame retardant was added, and the flame retardant was injected at a pressure of 2 MPa using a pressurized pump.

We evaluated the impregnation performance at the first 30 min and every hour. We weighed cedar to 120 min and larch to 720 min. Because, Japanese cedar is relatively well impregnated, Korean larch is an impermeable species, so the impregnation process took longer than Japanese cedar.

After the flame retardant on the sample surface was thoroughly wiped off, the sample weight was measured. The difference in weight was utilized in Eq. 5 to calculate the amount of impregnated flame retardant. In addition, after the impregnation process is completed, the wood samples were cut at 10 cm intervals to assess the impregnation weight of flame retardant for each length. Visual inspection was also conducted during this process:

$$R = \frac{m_2 - m_1}{V} \quad (5)$$

where R is the flame-retardant dosage (kg/m^3), m_1 is the sample weight before flame retardant impregnation (kg), m_2 is the wet sample weight after flame retardant impregnation (kg), and V is the sample volume (m^3).

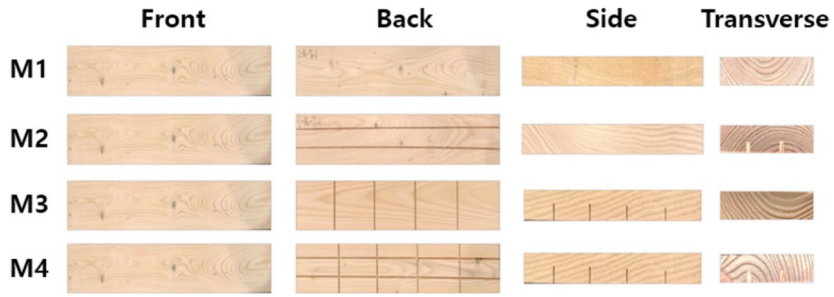


Fig. 1 Four types of grooving process model

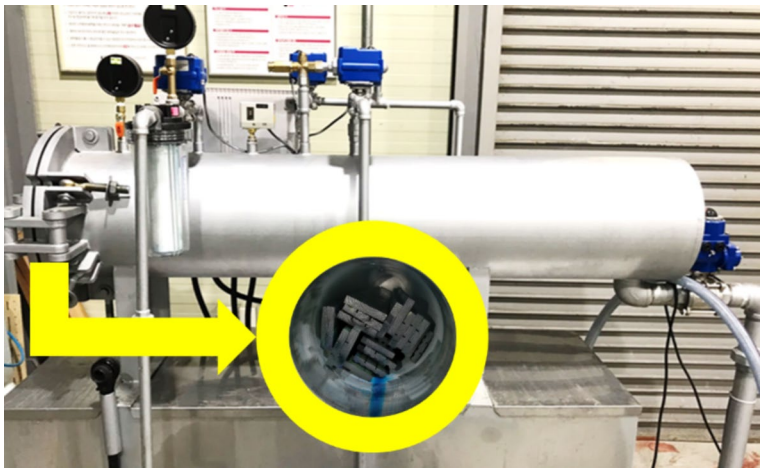


Fig. 2 Vacuum pressure impregnation chamber

Results and discussion

Porosity and gas permeability analysis

Figure 3 presents the porosity and permeability analysis results of Japanese cedar and Korean larch. The open-pore porosity of Japanese cedar was 75.5%, and Korean larch's was 55.6%. The result is that the open-pore porosity of Japanese cedar is 1.4 times greater than that of Korean.

Open pore is a mixed form of blind pore and through pore [23]. Gas permeability correlates with through pore. Therefore, a relative comparison of through pore porosity is possible by analyzing gas permeability [22, 23, 27, 29]. The gas permeability of Japanese cedar and Korean larch were 0.18 and 0.11 Darcy, respectively, indicating that Japanese cedar was approximately 1.6 times greater than Korean larch. As a result of analyzing the difference in open porosity and gas permeability between Japanese cedar and Korean larch by *T* test, the significance probability was within 0.01%, which was statistically significant. In light of these results, it can be assumed that Japanese cedar's flame-retardant impregnation performance is far superior to Korean larch's.

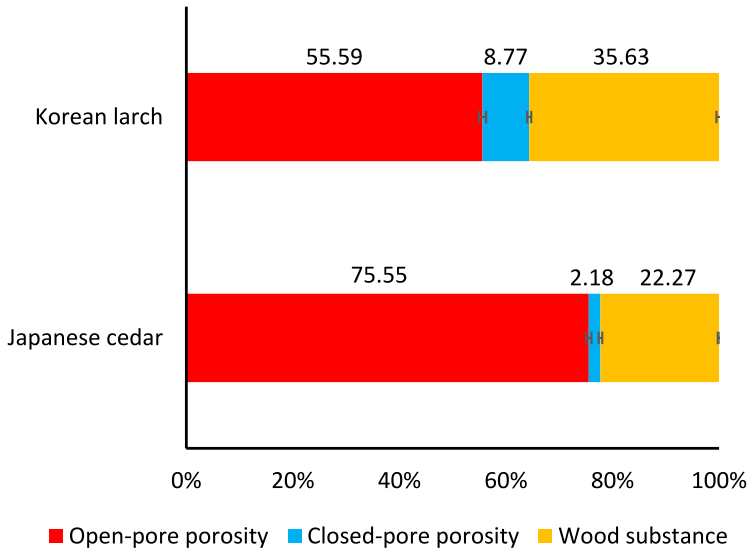
Impregnation performance

Figure 4 provides the flame-retardant dosage according to the grooving model. Figure 4a shows flame-retardant dosage of Japanese cedar, and Fig. 4b depicts that of Korean larch. The weight of the solution impregnated into the sample increased with increasing impregnation time. Japanese cedar had better impregnation performance than Korean larch [$p < 0.01$ by Duncan test (ANOVA)]. This is because Korean larch has lower open-pore porosity than Japanese cedar, so its permeability is not excellent. Anatomically, it is known that the main cause is the development of pit aspiration in Korean larch.

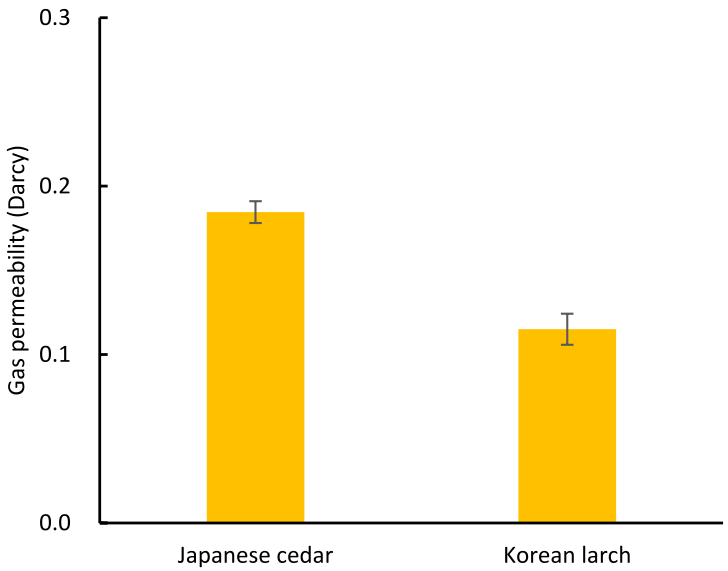
Japanese cedar's flame-retardant impregnation performance differed depending on the grooving model. After 120 min, the impregnation dosage per unit volume of M1 was 689.9 kg/m^3, while M2 was 770.9 kg/m^3, sample M3 was 797.7 kg/m^3, and sample M4 was 757.3 kg/m^3. The fiber direction grooved model (M2) showed about 11.7% improved impregnation performance compared to the unprocessed (M1). In addition, the model (M3) with grooves in the direction perpendicular to the fiber showed an improvement of about 15.6% compared to M1. However, M4 showed no statistically significant improvement. Basically, Japanese cedar was a species with excellent permeability. Although the grooved models showed a slight improvement in impregnation performance, no significant or dramatic difference was observed.

The flame-retardant impregnation performance of Korean larch also exhibited variations depending on the grooving model applied. After 720 min, the impregnation dosage per unit volume for M1 was 192.2 kg/m^3, M2 was 196.9 kg/m^3, M3 was 332.3 kg/m^3, and M4 was 360.3 kg/m^3. M1 showed no statistically significant difference from M2. However, M3, which incorporated grooves perpendicular to the fiber direction, exhibited a remarkable improvement of around 72.9% compared to M1. M4 showcased an impregnation performance enhancement of 87.4% compared to M1. However, there was no statistically significant difference in impregnation performance between M3 and M4.

Korean larch is known as a species with poor permeability. In this study, grooving in the fiber direction did not significantly affect impregnation performance improvement. On the other hand, grooving



(a) Porosity

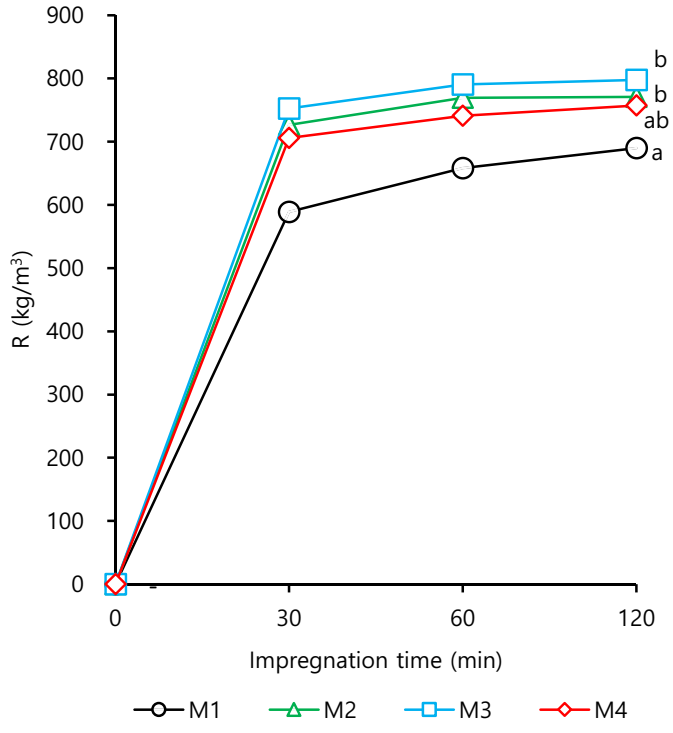


(a) Gas permeability

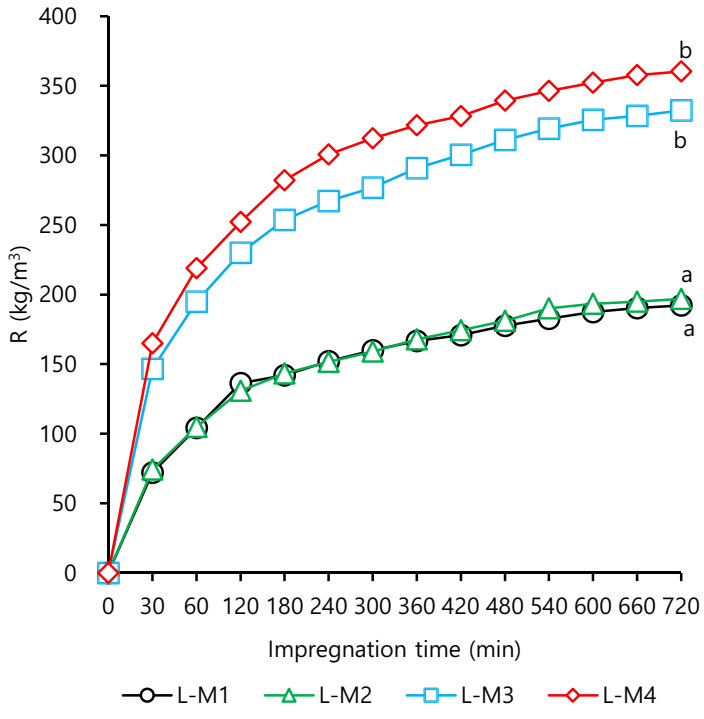
Fig. 3 Porosity and gas permeability analysis (error bars are standard deviation)

in the direction perpendicular to the fiber direction resulted in a dramatic enhancement of impregnation performance.

Figure 5 displays the comparison of impregnation performance in the longitudinal direction based on the type of grooving. Flame retardant materials can be impregnated through all sides of the wood. Still, since the permeability of the cross-section is superior to that of radial and tangential sections, the amount impregnated through tracheids in the fiber direction is the highest. Therefore, the amount of impregnation is

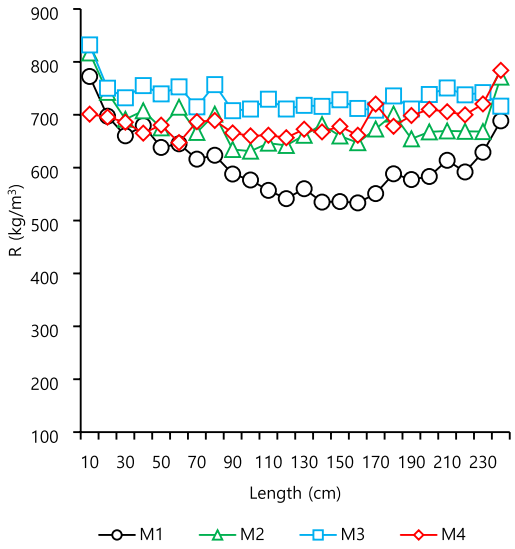


(a) Japanese cedar

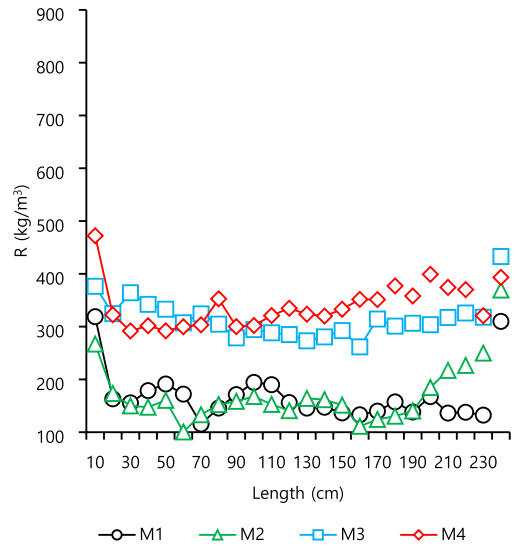


(b) Korean larch

Fig. 4 Flame-retardant dosage depending on the grooving model [sharing the same alphabet indicates no significant difference between the two groups (Duncan test, $p < 0.1$)]



(a) Japanese cedar



(b) Korean larch

Fig. 5 Comparison of impregnation performance in the longitudinal direction based on the grooving model

greater at both ends of the wood, and the impregnation performance decreases as it enters the interior.

Figure 5a shows the impregnation profile of Japanese cedar. The difference between the maximum and minimum impregnation weight for each part of M1 was 239.0 kg/m^3 (aqueous), 185.70 kg/m^3 (aqueous) for M2, 124.40 kg/m^3 (aqueous) for M3, and 136.50 kg/m^3 (aqueous) for M4. A small gap between the maximum and minimum impregnation weight means the flame retardant is evenly penetrated. As a result, it was confirmed that the grooving in the direction perpendicular to the fiber contributed more to the even penetration of the flame retardant than the grooving in the fiber direction.

Figure 5b illustrates the impregnation profile of Korean larch. The observed differences between the maximum and minimum impregnation weight for each grooving model were 203.30 kg/m^3 (aqueous) for M1, 268.80 kg/m^3 (aqueous) for M2, 171.20 kg/m^3 (aqueous) for M3, and 180.40 kg/m^3 (aqueous) for M4. The increased exposure of tracheids, characteristic of impermeable species, was found to facilitate more even penetration of the flame retardant.

Korea Ministry of Land, Infrastructure and Transport Notice No. 2020–1053 "Flame retardant performance of building finishing materials and fire diffusion prevention structures" stipulates that the total heat emission of semi-noncombustible materials should be 8 MJ/m^2 less for 10 min test. In addition, the total heat emission of flame retardant materials should be 8 MJ/m^2 less for 5 min test. Our previous study reported that the minimum impregnation of flame retardant for larch and cedar to fall within this standard of flame retardant material is approximately 94 kg/m^3 (solid) for Korean larch and about 145 kg/m^3 (solid) for Japanese cedar [41]. In light of the results of these preceding studies, it was found that cedar wood can sufficiently exhibit flame retardant performance only by the pressure impregnation process without the pretreatment process of grooving. On the other hand, for larch to pass the notification of the Ministry of Land, Infrastructure and Transport, it was found that the pretreatment process of M3 and M4 is essential.

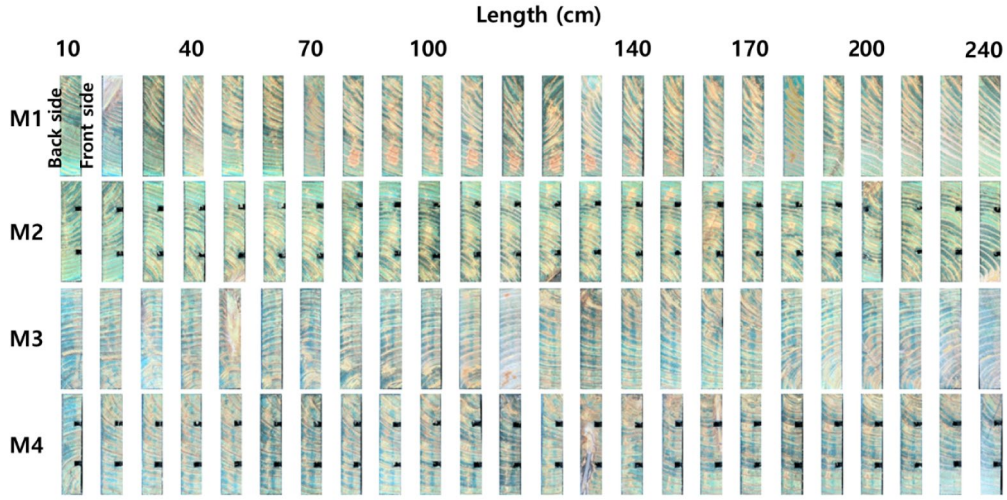
Visual inspection

Figure 6 represents the results of visual inspection of vacuum pressure impregnation in the fiber direction depending on the grooving model. Despite the significantly shorter impregnation time required for Japanese cedar compared to Korean larch, it has been confirmed that the impregnation performance of Japanese cedar is notably good.

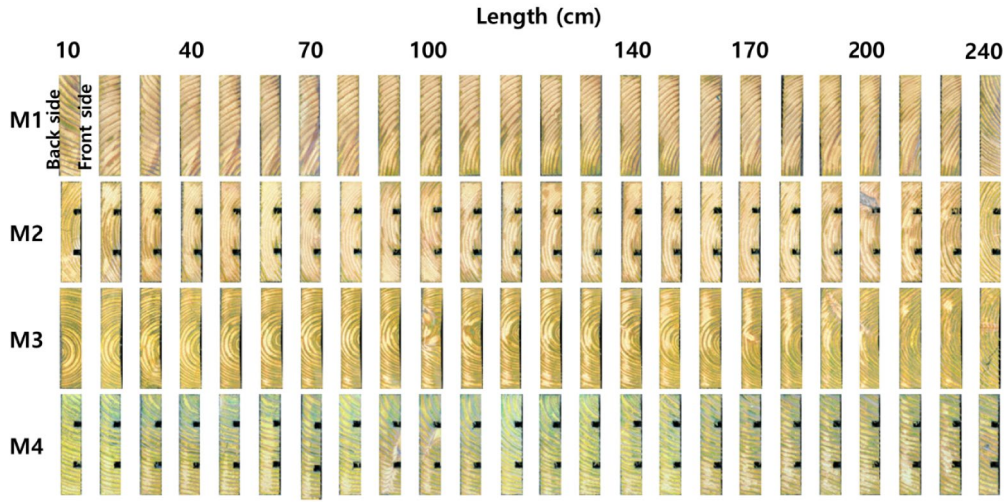
Overall, it was confirmed that grooving perpendicular to the fiber direction has advantages in terms of flame-retardant penetration compared to grooving along the fiber direction. Grooving pretreatment was a simple pretreatment method that did not require the introduction of special facilities and did not add complicated processes.

Conclusions

This study evaluated the effect of the groove processing method on the impregnation performance of commercial full-size Japanese cedar and Korean larch timbers used as building exterior materials. Since cedar is a species



(a) Japanese cedar (120 min)



(b) Korean larch (720 min)

Fig. 6 Visual inspection of vacuum pressure impregnation in the fiber direction depending on the grooving model

with excellent permeability, it did not show a dramatic increase in impregnation performance in groove treatment. On the other hand, Korean larch is a tree species with poor permeability, and groove processing in the vertical direction of the fiber dramatically improved the impregnation performance. In addition, the impregnation profile for each length confirmed that the grooving in the vertical direction of the fiber contributed more to the uniform penetration of the flame retardant than the grooving in the fiber direction. Through this study, we suggest that grooving pretreatment in the vertical direction of the fiber can improve the impregnation performance of impermeable commercial full-size timber.

Abbreviations

ρ_b	Density of specimen (g/cm^3)
ρ_t	True density of wood (g/cm^3)
ρ_p	Density of specimen using a gas pycnometer (g/cm^3)
D	Darcy permeability constant
F	Flow (cm^3/s)
T	Sample thickness (cm)
V	Viscosity of air (cp)
d	Sample diameter (cm)
P	Pressure (psi)
R	Flame-retardant dosage (kg/m^3)
m_1	Sample weight before flame retardant impregnation (kg)
m_2	Wet sample weight after flame retardant impregnation (kg)
V	Sample volume (m^3)

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Author contributions
ESJ: first author, writing (original draft), methodology, experiment, formal analysis, and writing (review and editing), SUJ: experiment and formal analysis, HJP: corresponding author, supervision, conceptualization, and writing (review and editing).

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Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
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