Preliminary Investigation on The Vacuum Pressure Impregnation Performance of Flame Retardant for Larch (*Larix kaempferi*) Depending on Grooving Type

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Due to wood's susceptibility to fire, it is crucial to treat wood-based materials with flame retardants, especially in construction applications. This study investigated the effectiveness of various grooving types, including transverse, longitudinal, both transverse and longitudinal, and surface grooving, in enhancing the vacuum pressure impregnation of larch wood. The results revealed that transverse grooving provided a slightly greater impregnation advantage than longitudinal grooving. Moreover, exceptional impregnation performance was observed in larch samples subjected to threefold longitudinal, transverse, and surface grooving, exhibiting a remarkable improvement of 215% compared to untreated larch. However, a limitation of this study is that only one wood species and one flame retardant formulation were used. While it is meaningful as a preliminary investigation into the vacuum pressure impregnation performance of flame-retardant wood based on groove processing, further studies using various wood species and flame retardants.

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INTRODUCTION

Wood has served as a fundamental construction material since ancient times (Eder *et al.* 2021). Compared to steel, concrete, and glass, the use of wood consumes relatively little energy during its manufacturing and disposal (Sandoli *et al.* 2021; Lin *et al.* 2023). Consequently, incorporating wood into construction supports the achievement of carbon neutrality (Scouse *et al.* 2020; Talvitie *et al.* 2021; Jang and Kang 2022b).

Notably, contemporary architecture has witnessed a rapidly growing interest in the construction of high-rise buildings using wood (Li *et al.* 2019). In 2019, an 85.4 m tall, 18-story multi-purpose timber building was completed in Brumunddal, Norway (WCN 2019). In 2022, an 86.5-m, 25-story wooden structure was completed in Milwaukee, Wisconsin. It is officially the tallest mass timber building in the world to date (USDA 2022). Furthermore, there is an ongoing project in Japan to construct a 70-story wooden skyscraper in Tokyo, reaching a height of 350 m. The building's inner frame is constructed entirely of timber, while the outer frame utilizes a hybrid structure. This combination ensures the structural integrity of the building, allowing it to withstand strong winds and earthquakes (Harada *et al.* 2020).

High-rise wooden buildings promote a sustainable building industry and contribute to resource conservation and environmentally friendly construction (Mirzaakbarovna and Sultanbayevich 2021). These structures are also regarded as artistic expressions that fuse the modern with the traditional.

Wood is inherently vulnerable to fire hazards, making fire safety a critical consideration in the design and use of wooden structures (Pabeliña *et al.* 2012; Jian *et al.* 2023). Therefore, flame retardant treatment is required to suppress, significantly reduce, or retard the combustion of wood. These treatment methods typically include surface treatment or coating with flame retardant chemicals and the impregnation of wood with chemical solutions or nanocomposites (Pabeliña *et al.* 2012; Wang *et al.* 2022).

This study focused on the vacuum pressure impregnation process for flame retardants. The vacuum pressure impregnation process is commonly known as the Bethel process or the full-cell impregnation process (Bryan 1932). This process impregnates the wood with chemicals in a sequence of steps including a vacuum, pressure, soaking time, drainage, and drying (Xu *et al.* 2020). Originally, this process was used for preserving wood, but it has also become widely used to impregnate wood with fire retardants (Rejeesh and Saju 2017; Xu *et al.* 2020).

The performance of the vacuum pressure impregnation process is affected by variables such as the species of wood, flow characteristics of chemicals, impregnation pressure, temperature, and impregnation time (Schneider *et al.* 2003; Yildiz *et al.* 2012).

Jang and Kang (2023b) investigated the effects of pressure, temperature, and time on larch (*Larix kaempferi*) and pine (*Pinus koraiensis*) wood impregnation using multiple regression analysis. Their study revealed that impregnation pressure had the most significant impact on the impregnation process, while time and temperature had comparatively less influence among the variables. This study suggests that controlling and optimizing the impregnation pressure can be crucial in achieving desired results in wood impregnation.

However, these methods alone have limitations in improving the pressure impregnation process for impermeable wood. Thus, physical pre-treatment, such as boring and incision, are widely used to enhance the vacuum pressure impregnation process for impermeable wood species (Yildiz *et al.* 2010; Park *et al.* 2017). Islam *et al.* (2008) applied laser incisions to Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco) under various conditions, and the optimum incision density was found to be 7,500 holes/m^2. Fukuta *et al.* (2022) reported that improved flame-retardant penetration was observed in wood pretreated by laser micro incisions with depths of 6 mm or less.

Park *et al.* (2017) conducted experiments involving kerfing and boring combinations under various conditions to improve the permeability of flame retardants. After comprehensively evaluating the permeability and the mechanical strength of the wood treated with flame retardant, they recommended a combination of 5 mm (D) x 3.5 mm (W) kerfing and boring with hole diameter of 12 mm or less.

Jang and Kang (2023a) introduced a steam explosion treatment as a means to improve the capabilities of wood following pressure impregnation. This treatment subjected the wood to steam and subsequent rapid decompression, which resulted in the creation of microcracks in the cell walls of the wood. This process led to an increased open pore content in the wood, ultimately enhancing the impregnation process. However, the physical properties of the wood treated with this method have not been identified, and there are limitations in applying this method to large-sized timber.

Hence, this study proposes a grooving process as a vacuum pressure impregnation pretreatment technique that can be readily implemented for large timber. This method involves creating grooves in the longitudinal and transverse directions. Transverse grooving can increase the exposure of the wood's tracheids compared to the longitudinal direction. We expect that the increased exposure of the tracheids will improve the permeability of the wood and thus improve the impregnation efficiency.

Hardwoods transport water and nutrients through vessels, and their pore size is relatively large compared to the pores within the structure of softwoods. Softwoods transport water mainly through the lumen spaces within tracheids, which are long and narrow and have low permeability. Among softwoods, larch has well-developed resin canals, and the pits of the tracheids have thick and complex structures, making it a species with low permeability (Kolya and Kang 2021; Song *et al*. 2022). Therefore, this study selected Korean larch *(L. kaempferi)* as a suitable tree species for a preliminary investigation into the vacuum pressure impregnation performance of flame retardants, depending on the grooving type.

EXPERIMENTAL

Specimen Preparation

This study used Korean larch, a representative impermeable wood, as the subject material. We sourced dried lumber from a domestic lumber supplier, and samples were cut to 1,030 mm (L) x 100 mm (W) x 19 mm (T). Their air-dried density was 0.57 g/cm^3 and their moisture content was 12.9%.

Grooving Process

Five types of samples were prepared to compare the efficacy of the flame retardant's vacuum pressure impregnation by grooving style. Sample A was the control group. Sample B was grooved longitudinally with a width of 3 mm and a depth of 7 mm. Sample C was grooved transversely. Sample D was grooved both longitudinally and transversely. Sample E was grooved both longitudinally and transversely and had additional surface irregularities. Figure 1 provides diagrams of the grooving types of the larch samples used in this study.

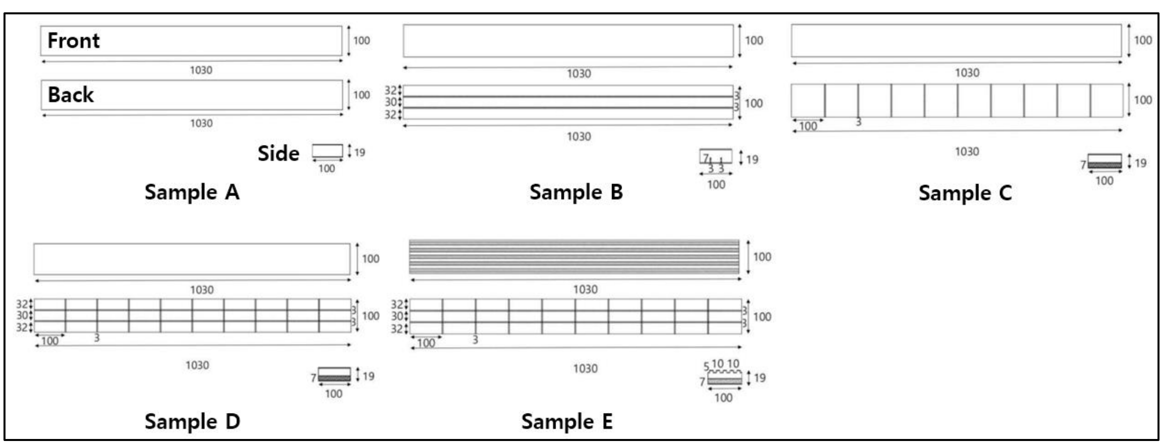

Fig. 1. Grooving process of Korean larch

Flame Retardant

This study used a water-soluble flame retardant (SafeWood Co Ltd., Korea), whose main ingredients were ammonium phosphate dibasic and additives. The resin content on a dry mass basis of each sample was about 27%, the specific gravity was 1.13, and the pH was 7.6. A 3% dilution of water-soluble blue ink was added to the flame-retardant formulation to easily check the permeability of the flame-retardant with the naked eye.

Vacuum Pressure Impregnation Process

This study utilized a wood vacuum pressure impregnation chamber that was developed in house at Jeonbuk National University. The specimens were placed in the chamber and depressurized at -0.1 MPa for 5 min using a vacuum pump. Then, the flame retardant was added while maintaining a vacuum in the chamber and pressurized at a pressure of 20 kgf/cm² (285 psi) for a range of times up to 720 min using a pressurization pump to inject the flame retardant into the test specimen. After the flame retardant was injected, the pressure was released, the flame remaining retardant was recovered, and the vacuum pump was operated to decompress the samples for 5 min to recover the flame retardant from the wood surface, completing the flame-retardant impregnation process.

Flame Retardant Impregnation Amount

The impregnated samples were collected every 30 min for the first 60 min and every 60 min thereafter until 720 min. Each specimen was weighed before and after flame retardant impregnation, and the difference was used to calculate the amount of impregnated flame retardant, as shown in Eq. 1,

$$D = \frac{m_2 - m_1}{V} \tag{1}$$

where D is the flame-retardant dosage (kg/m^3), m_1 and m_2 are the sample weight before and after flame-retardant impregnation (kg), respectively, and V is the sample volume (m^3). In addition, the samples were cut at intervals of 10 cm vertically to calculate the flame-retardant impregnation amount for each length, and visual inspection was also conducted.

RESULTS AND DISCUSSION

Impregnation Performance

Figure 2 provides impregnation performance depending on the grooving type. All five samples demonstrated that the flame-retardant impregnation amount increased as the impregnation time was increased. This is a typical trend for the vacuum-pressure impregnation process (Jang and Kang 2023a,b). The impregnated amount varied depending on the grooving type. At 720 min, the control (sample A) had an impregnation mass per unit volume of 168.3 kg/m^3, while it was 234.5 kg/m^3 for sample B, 237.5 kg/m^3 for sample C, 278.0 for sample D, and 361.3 kg/m^3 for sample E. The impregnation performance of the longitudinally grooved samples was enhanced by approximately 139.3% compared to the control, while the transversely grooved samples showed an improvement of 141.1%. The transversely grooved larch exhibited better impregnation performance due to increased exposure of tracheids, which have higher permeability in the cross-section of the wood. However, the difference between the grooving directions was just 1.8% due to the larch's pit aspiration. Pit aspiration in conifers is well-known as a cause of reduced permeability (Comstock and Côté Jr 1968; Bao *et al.* 2001).

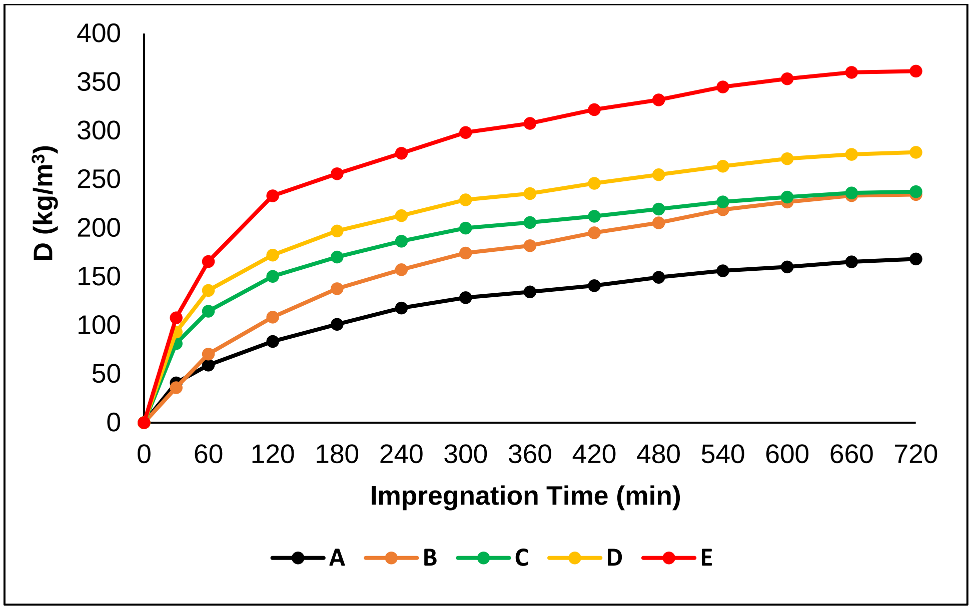

Fig. 2. Impregnation performance depending on the grooving type

When both transverse and longitudinal grooving was applied to the fibers, the impregnation performance was enhanced by around 165.2%. Furthermore, adding surface grooving processing to the longitudinal and transverse grooved sample resulted in a notable improvement of approximately 214.7%. This suggests that the surface grooving processing enhanced flame-retardant absorption by increasing the wood's specific surface area.

Figure 3 shows the impregnated amount in the longitudinal direction depending on the grooving type. All larch samples showed high flame-retardant impregnation at both ends but a gradual decrease in impregnation toward the center.

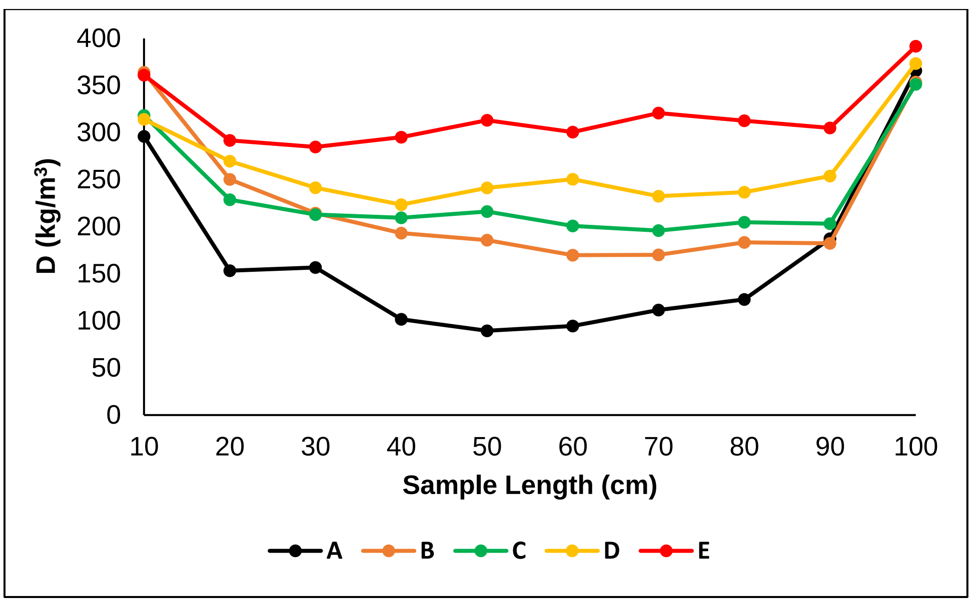

Fig. 3. Comparison of impregnation performance in the longitudinal direction depending on the type of grooving

The difference between the maximum and minimum impregnation amounts in the control group was 276.2 kg/m^3. It was 194.2 kg/m^3 for sample A, 194.2 kg/m^3 for sample B, 155.5 kg/m^3 for sample C, 150.0 kg/m^3 for sample D, and 106.9 kg/m^3 for sample E. As a result, the flame-retardant impregnated amount was more even in the transverse grooving than in the longitudinal grooving, and the flame retardant was more evenly impregnated when grooving in both the longitudinal and transverse directions and when surface roughness processing was added. As the exposure of tracheids increased and the surface area of the wood increased, the flame retardant was impregnated increasingly evenly.

Visual Inspection

Figure 4 displays the results of visual inspection of impregnation in the longitudinal direction depending on the grooving type. In the untreated wood, the flame retardant was impregnated only at both ends and barely penetrated the center. However, it was confirmed with the naked eye that the flame retardant penetrated evenly to the central part according to the additional groove processing. This shows the impregnated amount in the order of A<B<C<D<E following the same trend as Fig. 3.

Anatomically, fire retardants penetrate softwood through tracheids aligned in the direction of the fibers. However, due to the well-developed resin canals and the thick pits of the tracheids, penetration beyond a certain depth is challenging. The groove processing that was done in the present work helped evenly impregnate the wood with fire retardants to overcome this limitation.

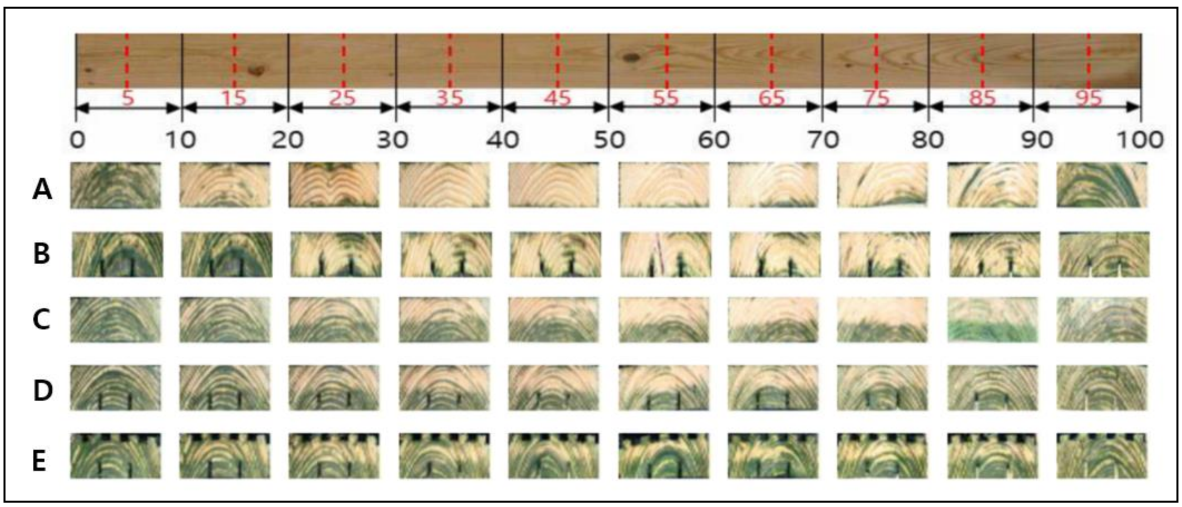

Fig. 4. Visual inspection of impregnation in the longitudinal direction depending on the grooving type

This study served as a preliminary investigation into the impact of grooving on enhancing larch's flame-retardant-impregnated amount. Future research will explore the alterations in physical properties resulting from groove machining. The aim will be to identify a groove processing technique that optimizes the impregnated amount while minimizing changes in the physical properties of the treated wood. The goal here is to incorporate not only new grooving processes but also a range of physical and chemical treatment methods to enhance the permeability of wood. Previous studies have reported that the permeability of wood can be improved through heat treatment, physical treatments such as ultrasound or microwave, and chemical treatments such as delignification. Accordingly, the plan is to conduct additional studies on enhancing the impregnation of flame retardants by applying these technologies (Jang and Kang 2022a, 2023c). Ultimately, this study aims to improve the flame-retardant impregnation process for larch, a species known for its low permeability.

In the future, this research will be expanded by applying the technology of additionally coating flame retardants, such as ammonium phosphate, onto wood impregnated with flame retardants under vacuum pressure. Furthermore, to apply this technology industrially, it is necessary to research the economic benefits of groove processing compared to other treatment methods for improving the flame retardancy of wood.

CONCLUSIONS

1. The study focused on utilizing grooving pretreatment processes to enhance the flame-retardant impregnation of Korean larch, a wood species known for its impermeable characteristics. The flame retardant was more effectively impregnated when the grooves were made in the cross-fiber direction rather than the longitudinal direction.
2. When groove processing was performed in both longitudinal and transverse directions for 720 min, and groove processing was added to the surface, this resulted in the highest flame-retardant impregnated amount. This approach yielded approximately 214.7% improved impregnated amount compared to untreated larch.

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Authors' Contributions

ESJ: First author, writing (original draft), writing (review & editing), XJK: experiment, formal analysis, writing (review & editing), SUJ: experiment, formal analysis, writing (review & editing), HJP: corresponding author, supervision, conceptualization, methodology, writing (review & editing).

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