Effect of Steam Explosion Treatment on Impregnation of Three Species of Softwoods: North American Spruce, Korean Pine, and Japanese Larch

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Effects of steam explosion were investigated relative to impregnation of wood. Three types of softwoods [North American spruce (*Picea orientalis*), Korean pine (*Pinus koraiensis*), and Japanese larch (*Larix kaempferi*)] were prepared and subjected to steam explosion treatment. The cross-sectional surfaces of the samples were observed with SEM, and their open-pore and closed-pore porosities were determined using a gas pycnometer. The softwoods were vacuum impregnated using ACQ-2 (Alkaline Copper Quaternary), a commercial preservative. After steam explosion treatment, the impregnation amount increased by 42.9% in the spruce and 155% in the Korean pine. However, there was no significant difference in the Japanese larch. The results from this study indicated that the steam explosion treatment helped to improve open-pore porosity by generating micro-cracks in the cell walls of softwoods, which improved the impregnation process. However, the degree of improvement in impregnation process differed by the species type.

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INTRODUCTION

Overcoming global warming will require substantial reductions in carbon dioxide (CO_2) emissions worldwide (Azadi *et al.* 2020; Deng *et al.* 2020). Trees efficiently pull CO_2 from the atmosphere and store it in wood, thus reducing its presence in the atmosphere (Werner *et al.* 2006; Russell and Kumar 2017). Historically, deforestation was assumed to be detrimental to the environment (Hughes 2011). However, the subsequent planting of new trees was found to benefit efforts to overcome global warming because older trees have a reduced carbon absorption capacity, whereas young trees act as vigorous carbon sinks (Ney *et al.* 2019).

Wood is an eco-friendly material with excellent durability and mechanical performance (Sandberg *et al.* 2017). Its flexibility as a building material allows it to be used as a structural material, interior material, or exterior material in the construction field (Werner *et al.* 2006). When wood is left outdoors, it is attacked by microorganisms, which slowly deteriorates it (Feist 1990). These characteristics of wood are advantages in terms of natural purification but disadvantages in terms of durability. For this reason, wood used in the outdoors must be preserved (Kim 2013).

There are several methods for preserving wood, including heat treatment (Candelier *et al.* 2016; Kim 2016), coating (Nejad and Cooper 2011), immersion (İlker 2021), and impregnation processes (Soulounganga *et al.* 2004). Of these, the most reliable method is impregnation, in which chemicals are forced into the pores of wood using vacuum or high pressure. Wood has different pore shapes, and its content varies depending on the species.

Therefore, to predict impregnation, the pore-filling ratio (open-pore porosity) by skeletal density is an important parameter (Wu *et al.* 2017). Generally, a higher open-pore porosity is associated with higher gas permeability (Jang and Kang 2019; Jang *et al.* 2020). Therefore, the impregnation process can be facilitated through pre-treatment that increases the permeability of wood (Lehringer *et al.* 2009).

Pretreatment methods for improving permeability include treatments using microwave (He *et al.* 2017), ultrasonic wave (Kang *et al.* 2021a), alkaline enzyme (Durmaz *et al.* 2015), delignification (Kang and Lee 2005; Jang and Kang 2021), and steam explosion methods (Kolya and Kang 2021a). This study focuses on steam explosion treatment, which creates high steam pressure in a short time and destroys the cell lumen of wood (Kolya and Kang 2021a,b).

The steam explosion treatment results in a more open pore structure of the wood, as a result increasing its gas permeability (Kolya and Kang 2021a,b). The main purpose of this study was to determine whether steam explosion pretreatment can improve the impregnation of wood.

EXPERIMENTAL

Sample Preparation

This study examined air-dried timbers of North American spruce (*Picea orientalis*), Korean pine (*Pinus koraiensis*), and Japanese larch (*Larix kaempferi*) that were approximately 25 years old (Jeon-il Timber Co., Ltd; Gimje, Jeollabuk-do, Korea). After the timbers were cut to sample sizes of 2 × 2 × 29 cm, 10 intact samples without cracks and knots were selected from each species. The moisture content was 7.2% for spruce, 7.6% for Korean pine, and 7.3% for Japanese larch, as measured by KSF-2198 (2001). The densities of the samples were 0.52 ± 0.03 for spruce, 0.40 ± 0.03 for Korean pine, and 0.54 ± 0.06 for Japanese larch, as measured by KSF-2199 (2016) . The samples were cut in half and divided into control and steam explosion treatment groups. Figure 1 shows the samples from the three softwood species prepared for this study.

Steam Explosion Treatment

Samples from the three softwood species in the steam explosion treatment group were immersed in ultrapure water first and then decompressed at -0.1 MPa in a vacuum chamber for 4 weeks. Figure 2-a shows the schematic representation of the steam explosion treatment. A steam explosion machine (Model CK-0533579092, Chilgok, Korea) was used, as in previous studies (Kang *et al.* 2021b; Kolya and Kang 2021a,b). This machine was designed originally for production of puffed rice snacks; however, in this study, it was used for steam explosion of wood.

First, water-saturated specimens were sealed in the chamber. When the lower part of the chamber was heated, the pressure in the chamber increased. After approximately 13 min, the pressure had been increased to 10 bar. Then, the operator opened the chamber to release the steam. After that, the specimens were collected and naturally dried in the laboratory at 20 °C for 2 weeks. The steam explosion treatment was conducted by an operator from a Korean traditional puffed rice store (Moraenae puffed rice, Jeonju. Korea).

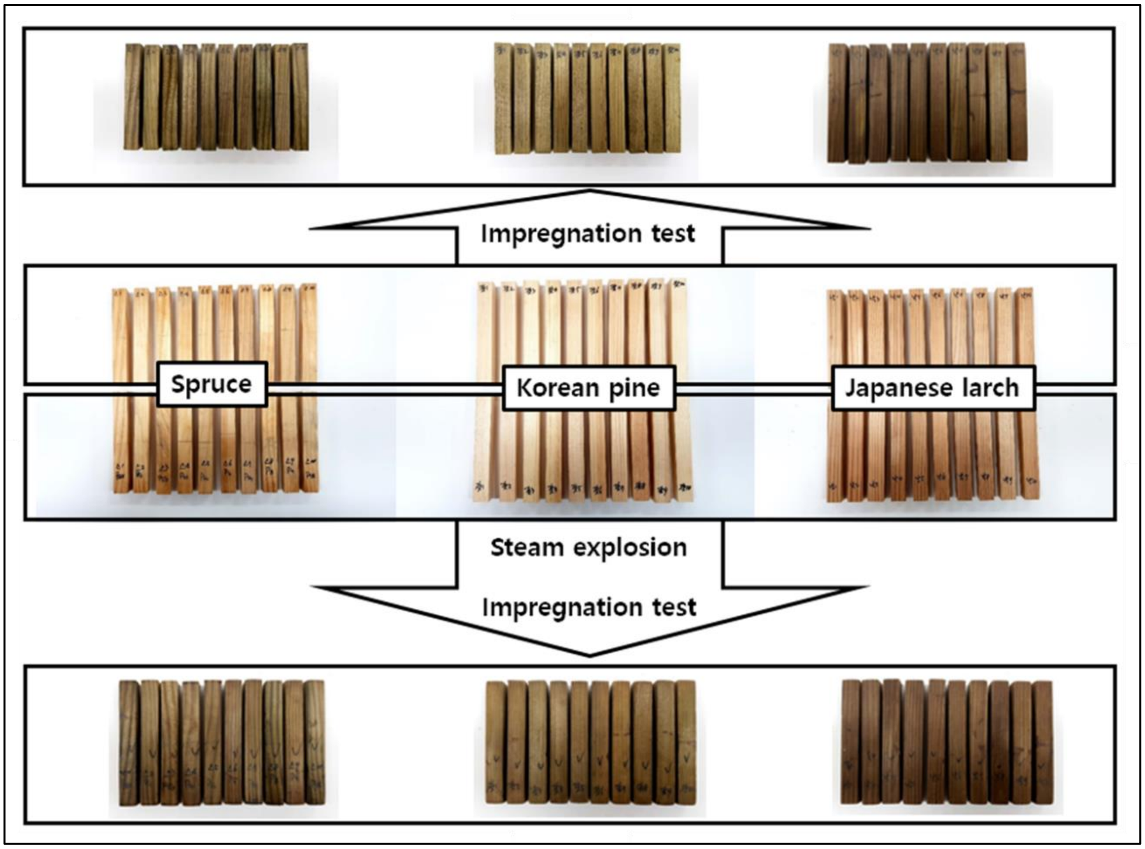

Fig. 1. Sample preparation for steam explosion and impregnation

Cross-sectional Morphology Before and After Steam Explosion

To observe the cross-sectional surfaces of the three softwoods before and after steam explosion, SEM analysis (Genesis-1000, Emcrafts, Korea) was used. The softening, microtome cutting, drying, and ion-coating processes were performed according to the reported pretreatment method in the SEM measurement of wood specimens (Jansen *et al.* 1998). The SEM analysis used 15 KV in high vacuum mode and 1,000× magnification for observation.

Analysis of Porosity and Open-Pore Porosity

Each sample density ρ (g/cm^3) was measured by its weight and volume. Sample porosity ϕ_t (g/cm^3) was calculated from wood substance density and sample density (Eq. 1). According to previous studies, the wood substance density ρ_{ws} (g/cm^3) was assumed to be 1.50 g/cm^3 (Lindgren 1991; Moore *et al.* 2007; Tanaka *et al.* 2014).

$$\phi_t\ (\%) = \left(1 - \frac{\rho}{\rho_{ws}}\right) \times 100 \quad (1)$$

Open-pore porosity (ϕ_o) can be analyzed by gas pycnometry per ISO 12154 (2004) . The skeletal density of each sample was obtained through a gas pycnometer (PYC-100A-

1, PMI, USA), and the porosity was calculated using Eq. 2:

$$\phi_o\ (\%) = \left(1 - \frac{\rho}{\rho_s}\right) \times 100 \quad (2)$$

The closed-pore porosity (ϕ_c) was calculated by subtracting the open-pore porosity from the total porosity, as in Eq. 3:

$$\phi_c\ (\%) = \phi_t - \phi_o \quad (3)$$

Impregnation

Figure 2-b shows a schematic representation of the impregnation test used in this study. Specimens were placed in a water bath. Then impregnation solution was added, and a heavy mesh was used to prevent the specimens from floating. The impregnation solution was prepared by diluting a commercial outdoor wood preservative (ACQ-2, Jeonil Timber Co. Ltd., Jeonju, Korea) at 8% (w/v) in distilled water (Ra *et al.* 2017; Pang *et al.* 2017).

ACQ-2 is widely used as a wood preservative for impregnation in Korea. It is composed of CuO 8.0% and dodecyldimethyl-ammonium chloride <8.0%. Typically, when impregnation treatment is finished, the specimen is left for about 10 minutes to recover the surplus ACQ-2. Then, the active preservative is cured for settlement in the treated wood for 4 weeks at room temperature (Kim *et al*. 2015).

In this study, impregnation was performed in a laboratory vacuum oven (model: OV4-30, Jeotech, Korea). The vacuum oven was depressurized to -0.1 MPa at 25 °C. Weights were measured every hour up to a maximum of 4 h. The amount of impregnation was calculated as in Eq. 4.

$$I = \frac{m_2 - m_1}{V} \quad (4)$$

Here, I represents the impregnation amount (g/cm^3), m_1 is wood mass before impregnation (g), m_2 is wood mass after impregnation (g), and V denotes wood volume (cm^3).

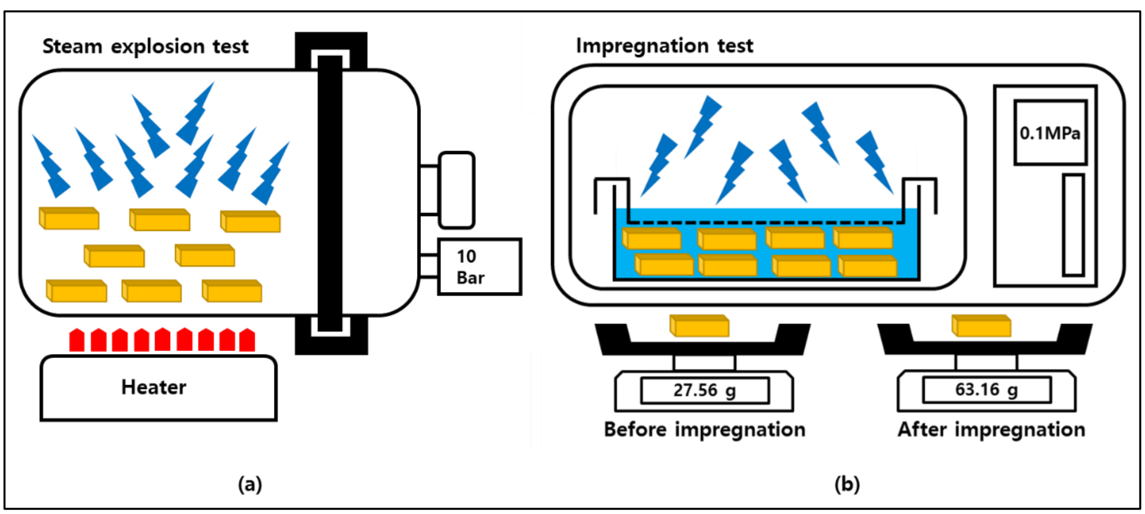

Fig. 2. Schematic diagram of the steam explosion chamber and impregnation test

RESULTS AND DISCUSSION

SEM Images of Cross-Sectional Surfaces

Figure 3 shows the SEM images of the cross-sectional surfaces of the three softwood specimens before and after steam explosion. After steam explosion, the cell walls of the spruce and Japanese larch were thinned and appeared flabby. In the Korean pine, a number of cracks were apparent in the cell wall.

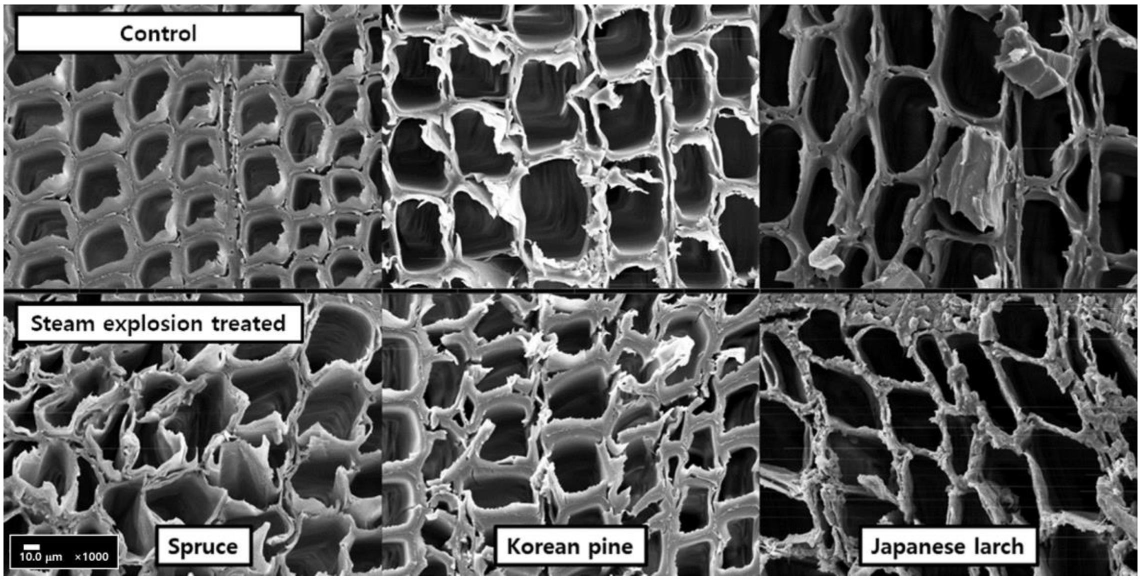

Fig. 3. SEM images of the cross-sectional surfaces of the three softwood specimens before and after steam explosion

Porosity Analysis

Figure 4 shows the open-pore and closed-pore porosities before and after steam explosion treatment. Before steam explosion, the open-pore porosity of spruce was 68.11 ± 0.76 % (± standard deviation), Korean pine was 43.10 ± 0.72%, and Japanese larch was 14.76 ± 0.38%. After steam explosion treatment, the open-pore porosity of spruce was 72.19 ± 0.38 %, Korean pine was 54.84 ± 0.29 %, and Japanese larch was 16.94 ± 0.39 %. The resulting increases were 6.0 % ($t = -14.11$, $P < 0.001$, paired T-test, $n=20$) for spruce, 24.9% ($t = -25.40$, $P < 0.001$, $n=20$) for Korean pine, and 15.5% ($t = -10.36$, $P < 0.001$, $n=20$) for Japanese larch, respectively after steam explosion treatment.

The pore structure changes and the improvements to the impregnation process for the three softwood species were observed. Previous studies (Kolya and Kang 2021a,b) reported that steam explosion of wood caused changes in the cellulose crystallinity of wood. Also, it can be assumed that the presence of rays (ray parenchyma cells) and resin canals changed to an open structure due to the steam explosion. These effects affect the increase in permeability. Modification of wood cell walls can be effective at improving air permeability. The results from this study showed that steam explosion caused micro-cracks in the cell walls, and the pore structure of the wood was opened.

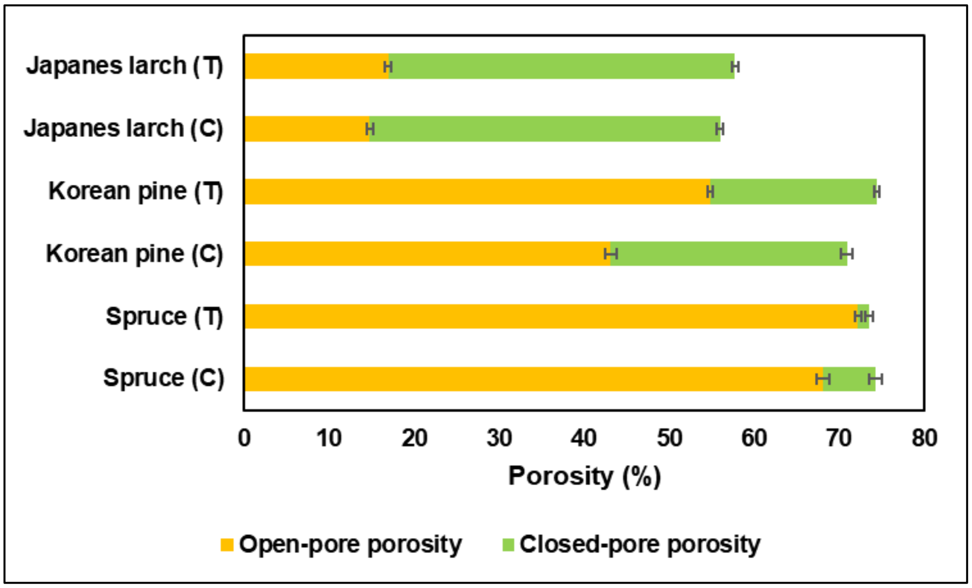

Fig. 4. Porosity analysis before and after steam explosion treatment (C: control, T: steam explosion treated)

Results of Impregnation

Figure 5 shows the impregnation amount before and after steam explosion for each of the softwood species examined. The impregnation patterns of the three species indicated that the wood samples were intensively impregnated during the first hour and then gradually increased thereafter. The impregnation amount was greatest for spruce, followed by Korean pine and Japanese larch. In summary, the impregnation performance of wood species is related to their open-pore structure but not linked to their porosity (Wu *et al.* 2017).

After one hour of steam explosion, the impregnation amount of Japanese larch increased by approximately 42.9% ($t = -4.733$, $P < 0.001$, n=20), from 0.07 to 0.10 g/cm^3. That of Korean pine increased by approximately 155 % ($t = -6.928$, $P < 0.001$, n=20), from 0.20 to 0.51 g/cm^3. However, spruce did not show a statistically significant increase ($t = 0.898$, $P = 0.392$, n=20).

After steam explosion, the Korean pine had the greatest increase in open-pore porosity among the three species. Accordingly, Korean pine showed the best impregnation performance of the three species examined. Conversely, Japanese larch had the lowest open-pore porosity among the three species. Although the open-pore porosity increased after steam explosion, it remained at a low level and was presumed to not affect the impregnation process. Accordingly, no improvement in impregnation was found in Japanese larch after steam explosion.

Permeability has a decisive influence on impregnation. There are various methods for improving the permeability of wood. Heat treatment effectively improves permeability, but processing time is long and energy consumption is high. The chemical treatment method requires chemical treatment and disposal facilities and might not be environmentally friendly. On the other hand, steam explosion treatment has the advantage of a simple facility and can change the wood's pore structure to an open type in a relatively short time.

As the result of this study, steam explosion changed the pore structure of the wood to be more open, which improved the gas permeability. This open pore structure helped to improve impregnation. However, steam explosion treatment may reduce strength. So, it is necessary to establish steam explosion treatment conditions that minimize the decrease in strength in the future. These results suggest that various parameters should be selected for steam explosion treatment based on the species. Future studies should evaluate patterns in impregnation resulting from the steam explosion in additional species, which can help to improve the impregnation process for the wood industry.

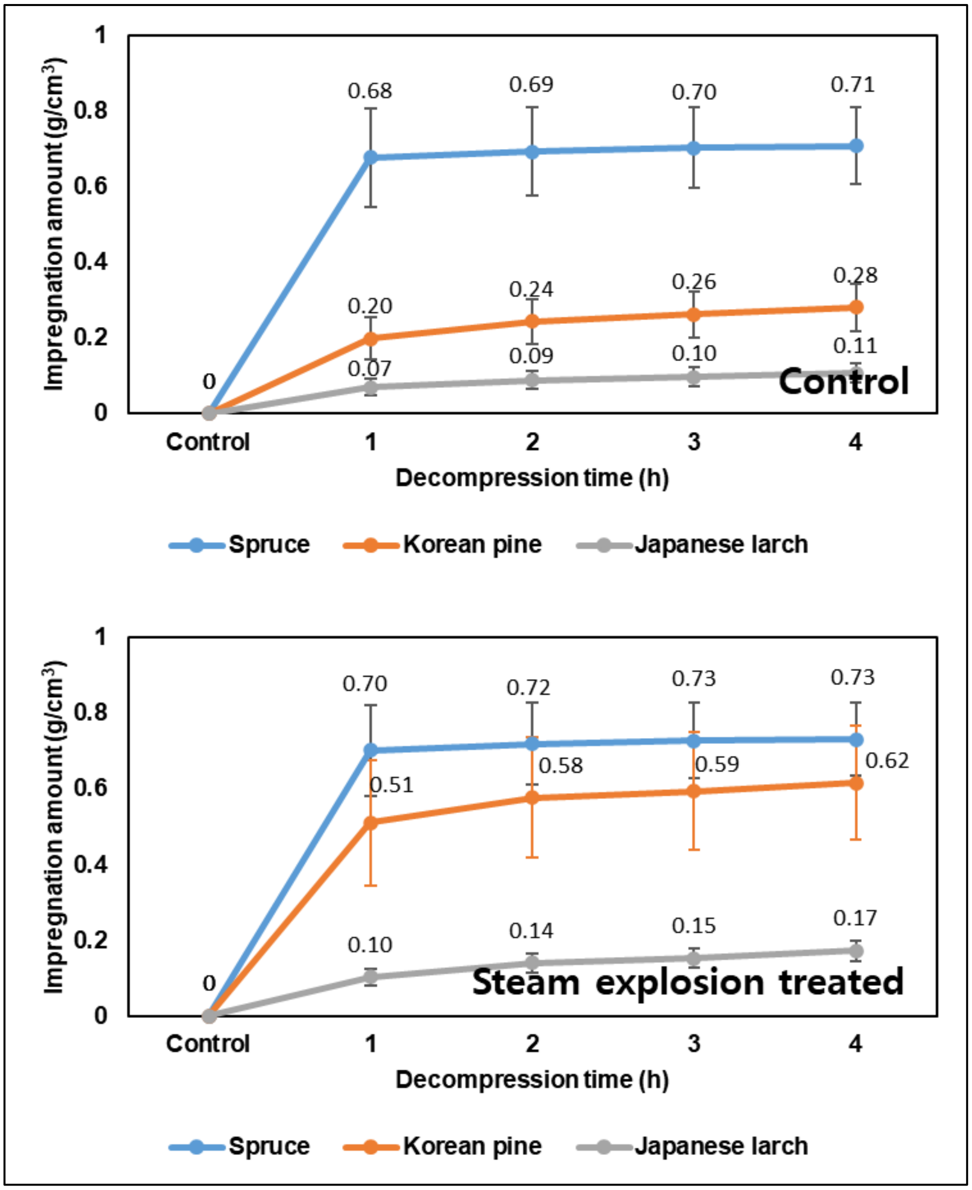

Fig. 5. Difference in impregnation amount before and after steam explosion treatment

CONCLUSIONS

1. After steam explosion treatment, the open-pore porosity of each species increased: 6.0% for spruce, 24.9% for Korean pine, and 15.5% for Japanese larch.
2. After steam explosion treatment, the impregnation amount increased by 42.9 % for spruce and 155% for Korean pine. However, Japanese larch did not show any significant improvements.
3. Steam explosion treatment helped improve open-pore porosity by creating microcracks in the cell walls, changing ray parenchyma cells, and resin canals softwoods.

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Authors' Contributions

Eun-Suk Jang is the first author of this study. He designed the study, conducted all experiments, and was a major contributor in the original writing, reviewing, and editing of the manuscript. Chun-Won Kang is the corresponding author, he was the supervisor of this project and contributed by reviewing and editing. All authors read and approved the final manuscript.

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