Effects of Pressure and Temperature on Wood Impregnation – Focusing on Larch (*Larix kaempferi*) and Korean Pine (*Pinus koraiensis*)

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The primary purpose of this study was to delineate the most significant factors in the wood impregnation process. For this, the authors prepared larch (*Larix kaempferi*) and Korean pine (*Pinus koraiensis*) wood and used alkaline copper quaternary as an impregnation solution. The chamber temperature was adjusted to 25, 50, and 80 °C, and the pressure was adjusted to 100, 200, and 300 psi. The impregnation process was maintained for up to 90 min. Multiple regression analysis was used to investigate the effects of temperature, time, and pressure in terms of amount of solution impregnated during the process. Factors affecting the amount of solution impregnated were as follows: the effect of pressure was greater than that of time, which was greater than that of temperature. Therefore, pressure was the most critical factor in the wood impregnation process.

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INTRODUCTION

Since prehistoric times, wood has been one of the most important natural materials for architectural applications (Asdrubali *et al.* 2017; Kuzman *et al.* 2018). In recent years, industrial activity involving building materials has been affected by environmental changes, underscoring the need for a sustainable approach (Luo *et al.* 2018; Orsini and Marrone 2019). The production of wood building materials uses less energy and emits less carbon than the production of reinforced concrete materials (Sathre and Gustavsson 2009). Accordingly, social interest in sustainability has led to an increase in the market of wood construction and products (Werner and Richter 2007).

Wood, however, has considerable disadvantages in terms of durability, biological properties, and stability under certain conditions (Jinzhen 2006; Kim 2013). The use of wood in outdoor applications requires preservative treatment to protect the wood from biological damage (Jinzhen 2006; Kim 2013). The preservative treatment of wood has a long history; the early settlers who migrated to the New World in the 17th century used preservatives to protect their wooden houses and other structures (Freeman *et al.* 2003).

Methods of preserving wood include brushing, spraying, soaking, and impregnation under pressure or vacuum (Teng *et al.* 2018). The impregnability of wood is affected by various parameters, *e.g.*, moisture content, drying method, and chemical structure of the preservative, but it is strongly related to its porous structure (Tarmian *et al.* 2020).

Ding *et al.* (2008) investigated the mechanisms of wood impregnation *via* methyl methacrylate (MMA) using a mercury porosimeter. They reported that the porosity of wood affects the impregnation rate when the wood pore diameter is greater than 0.1 µm. Wu *et al.* (2017) investigated the skeletal density of wood using a gas pycnometer and reported that the skeletal density and pore-filling ratio were proportional directly to lumen impregnation.

The permeability of wood reflects its pore structure. Wood with excellent permeability has an open pore structure (Jang *et al.* 2020; Jang and Kang 2021). Therefore, to improve the impregnation process, there are various biological, chemical, and mechanical methods that modify the pore structure of wood to increase permeability (Tarmian *et al.* 2020).

Konopka *et al.* (2018) suggested that, when measuring the moisture content (MC) of full-cell impregnated wood, the resistive moisture content measurement method is not suitable, and application of this method requires a modified formula.

Kumar (2021) investigated the effects of various compression parameters such as compression rate (CS), compression cycle (CC), compression ratio (CR), and compression direction (CD) on preservative absorption. It was observed that the compression ratio (CR) had the largest influence on liquid uptake of all the compression parameters. Also, absorption increased with increasing compression cycle (CC), whereas the direction of compression (CD) did not significantly affect absorption. Kumar proposed to improve the absorption of preservatives in lumber using compression.

In this study, the authors focused on the variables controlled during the wood impregnation process. For this study, larch and Korean pine, which are useful domestic species, were prepared. The amount of impregnation was measured at various temperatures, times, and pressures in the impregnation chamber.

The authors used an alkaline copper quaternary for this study, a material widely used as a wood preservative (Can *et al*. 2020).

Multiple regression analysis was performed with the impregnation amount as the dependent variable and time, temperature, and pressure as independent variables. The purpose of this study was to identify which time, temperature, and pressure had the greatest influence on the amount of wood impregnation.

EXPERIMENTAL

Sample Preparation

Timber from approximately 25-year-old Japanese larch (*Larix kaempferi*) and Korean pine (*Pinus koraiensis*) sapwood 32 (L) × 2 (R) × 2 (T) cm was supplied by Jeonil Timber Co., Ltd (Gimje, Korea). The wood was air-dried to a moisture content of 11%. The density was 0.59 g/cm^3 for Japanese larch and 0.74 g/cm^3 for Korean pine. Their annual rings numbered 6 to 7.

Forty-five samples of each species without cracks or knots were selected and divided into five groups of five samples each for treatment with different impregnation pressures and temperature conditions. Then, the volume and weight of each sample were measured.

A commercial outdoor wood preservative, alkaline copper quaternary (ACQ-2: 66.7% CuO and 33.3% dodecyldimethylammonium chloride), was supplied by Dae Heung

Chemical (Ansan, Korea) as an impregnation solution.

The active ingredient content of ACQ-2 solution was 16%, and the solution was prepared as a dilution at an active ingredient concentration of approximately 8% with distilled water.

Impregnation Treatment

A laboratory impregnation chamber was fabricated for this study, as shown in Fig. 1. The inner diameter of the sample chamber was 10 cm, and the depth was 70 cm. After placing five samples in the chamber, ACQ-2 solution was injected. The wood specimens were pressed into a mesh to prevent floating. The sample chamber was equipped with a double cover; the inner cover was sealed with an O-ring, and the outer cover was sealed with a screw thread. Impregnation pressure was achieved *via* an injection of nitrogen from a cylinder and was set to 100 psi (0.69 MPa), 200 psi (1.38 MPa), and 300 psi (2.07 MPa) using a gas regulator (model: HPS4-750-22R-4F, VICTOR, Junction, MD). In addition, the temperature was maintained with a thermostat at 25, 50, and 80 °C. The impregnation time was set to 90 min, and the amount of impregnation was measured every 30 min according to Eq. 1,

$$G = m_1 - m_0 \quad (1)$$

where G is the impregnation amount (g), m_0 is the sample weight before impregnation (g), and m_1 is the sample weight after impregnation (g).

The ACQ-2 preservative retention was calculated according to Eq. 2,

$$R = \frac{GC}{V} \times 10\ kg/m^3 \quad (2)$$

where R is the preservative retention (kg/m^3), C is the amount of preservative in 100 g of the treatment solution (g), and V is the volume of sample (g/cm^3).

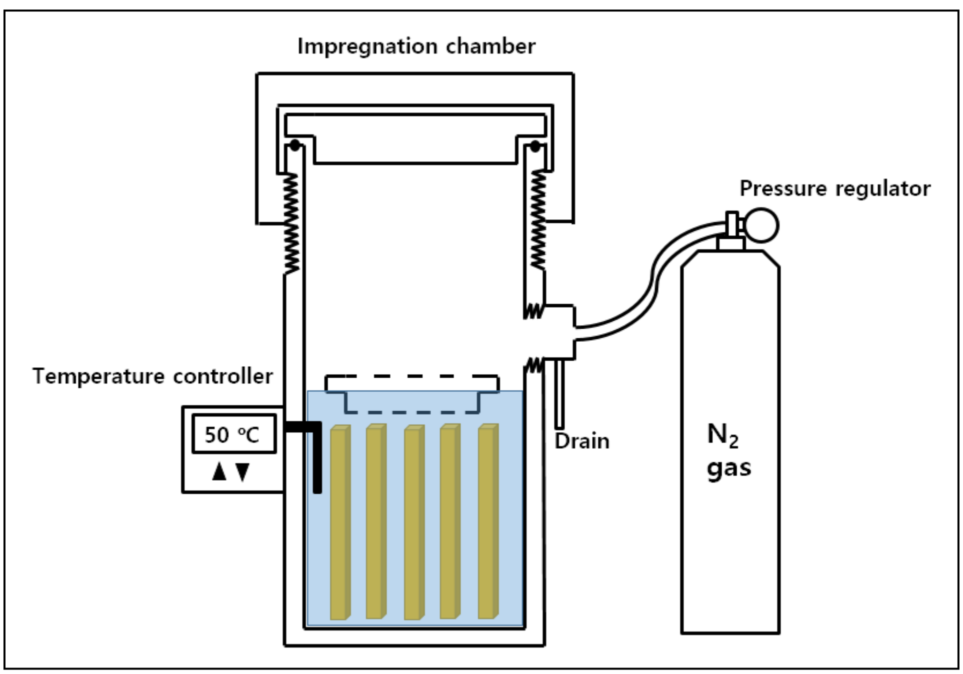


Fig. 1. Schematic diagram of the impregnation chamber

Statistical Analysis

The impregnation process requires user control of the pressure, time, and temperature. Therefore, this study aimed to statistically investigate to what extent these variables affect the impregnation process.

First, the effect of pressure on impregnation amount under each temperature condition was investigated. For this purpose, one-way ANOVA was used. The Duncan test was applied as the post hoc analysis method (Hilton and Armstrong 2006). The statistical correlation was analyzed *via* the Pearson correlation analysis to understand the univariate correlations between the time, temperature, and pressure of impregnation amount. Based on this, multiple regression analysis was performed.

Multiple regression analysis is a statistical method that includes multiple variables (factors) in one regression model and investigates whether these multiple variables simultaneously affect the dependent variable (Hamaker 1962). During wood impregnation, the time, temperature, and pressure variables are applied simultaneously. Therefore, this study investigated the effects of temperature, time, and pressure of impregnation treatment on liquid retention *via* multivariate regression analysis. The multivariate regression model is shown in Eq. 2,

$$Y = \alpha_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 D_1 + \varepsilon \qquad (2)$$

where Y is the retention, X_1 is the temperature, X_2 is the time, X_3 is the pressure; D_1 is the wood dummy variable (1: If the wood is larch, otherwise 0), α_0 is a constant (intercept term), and ε is the residuals (error term)

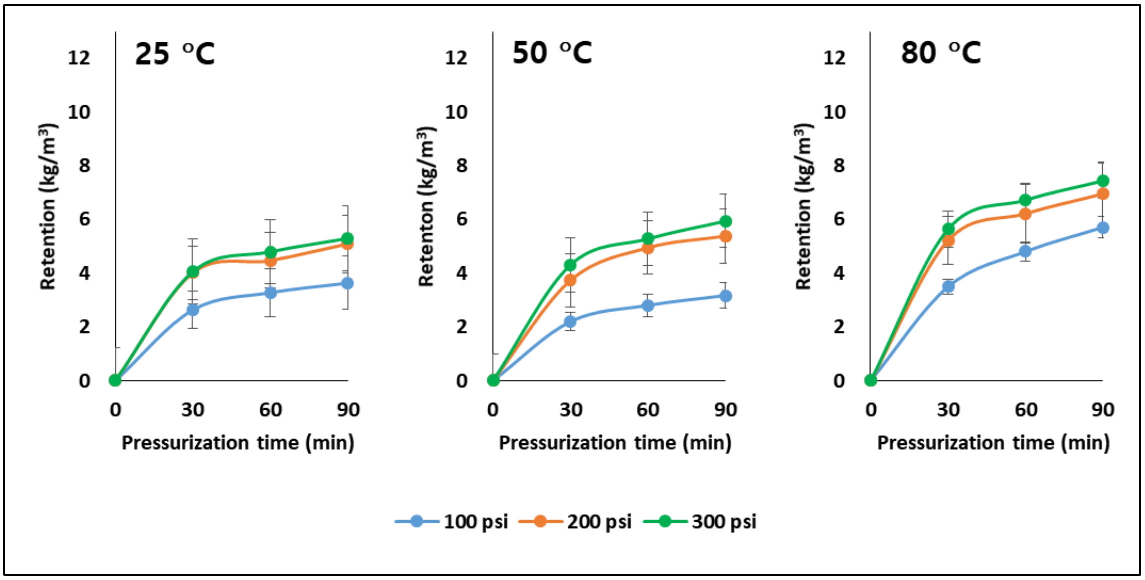
RESULTS AND DISCUSSION

Retention of Preservatives

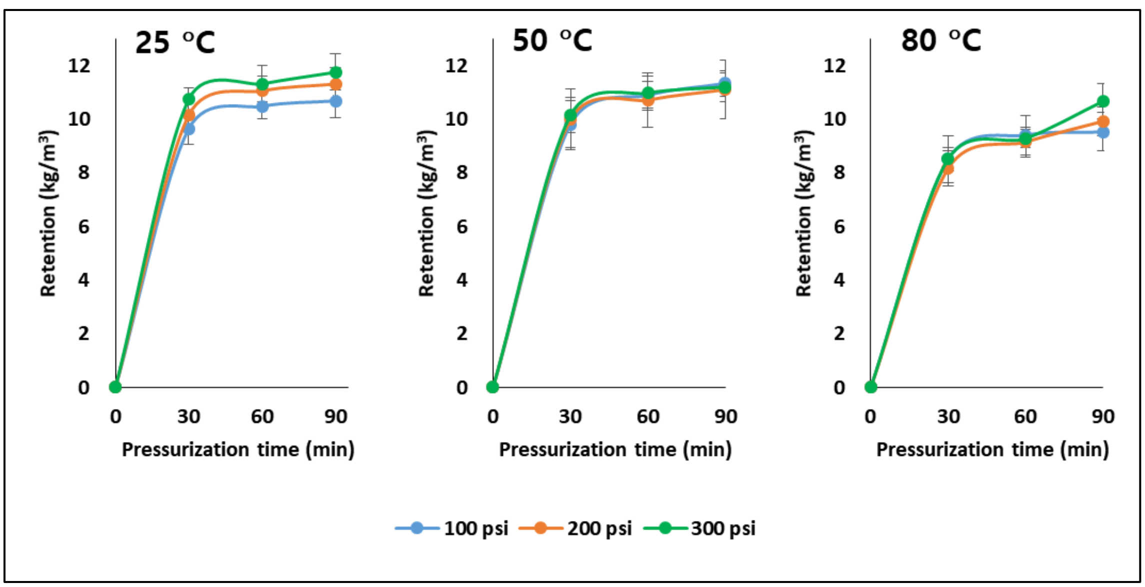
Figure 2 shows the retention for larch and Korean red pine with respect to pressure at process temperatures of 25, 50, and 80 °C. The overall pattern of the retention graph showed rapid impregnation for the first 30 min and a gradual increase thereafter.

Table 1 provides the retention of the preservatives of larch and Korean pine depending on pressure, time, and temperature. All results of the ANOVA had a p-value less than 0.1, which implied a statistically significant difference between the three groups according to pressure 100 psi (0.69 MPa), 200 psi (1.38 MPa), and 300 psi (2.07 MPa), at a significance level of 10%. The same letter indicates no significant difference, and different letters indicate a statistical difference at the 10% significance level *via* the Duncan test. In larch, the difference in retention with increasing time was not statistically significant at 100 or 200 psi pressure. However, there was a statistically significant difference in retention between 30 and 90 minutes at 300 psi pressure. In Korean pine, retention increased up to 60 min when the impregnation time increased at all pressures, and there was no significant difference between 60 min and 90 min.

Table 2 provides the results of the Pearson correlation analysis to determine the individual effects of temperature, time, and pressure on impregnation amount. Time and pressure showed a positive (+) correlation with impregnation amount at a significance level of 5%. However, the degree of correlation was larger for pressure (0.148) than time (0.120). In addition, the temperature did not show a significant correlation with impregnation amount. During wood impregnation, pressure was the most critical factor in determining impregnation amount.



(a)



(b)

Fig. 2. The retention graph with different temperatures and pressures for (a) larch and (b) Korean pine

Table 3 shows the results of the multiple regression analysis with impregnation amount as the dependent variable and temperature, time, and pressure as the independent variables. The F-Value of all the multiple regression models showed significance at 1%, which indicated strong statistical significance. In addition, the variance inflation factor (VIF) of all regression coefficients was less than 10, which meant that there was no multicollinearity problem between the regression models (Jang *et al.* 2020). The adjusted R^2 of the model was 0.946, which indicated an explanatory power of 94.6%.

Table 1. Results of Retention Depending on Temperature, Pressure, and Impregnation Time

Species	Temp (°C)	Pressure (psi)	Retention (kg/m^3) at Impregnation time (min)		
			30	60	90
Larch	25	100	$^{A}2.63^{a}$	$^{A}3.27^{a}$	$^{A}3.63^{a}$
		200	$^{A}4.01^{b}$	$^{A}4.47^{b}$	$^{A}5.09^{b}$
		300	$^{A}4.06^{b}$	$^{AB}4.80^{b}$	$^{B}5.29^{b}$
	50	100	$^{A}2.20^{a}$	$^{A}2.80^{a}$	$^{A}3.17^{a}$
		200	$^{A}3.73^{b}$	$^{A}4.95^{b}$	$^{A}5.38^{b}$
		300	$^{A}4.31^{b}$	$^{AB}5.28^{b}$	$^{B}5.94^{b}$
	80	100	$^{A}3.50^{a}$	$^{A}4.81^{a}$	$^{A}5.71^{a}$
		200	$^{A}5.22^{b}$	$^{A}6.22^{b}$	$^{A}6.95^{b}$
		300	$^{A}5.64^{b}$	$^{AB}6.72^{b}$	$^{B}7.44^{b}$
Korean pine	25	100	$^{A}9.64^{a}$	$^{B}10.47^{a}$	$^{B}10.68^{a}$
		200	$^{A}10.14^{ab}$	$^{B}11.05^{ab}$	$^{B}11.30^{ab}$
		300	$^{A}10.74^{b}$	$^{AB}11.31^{b}$	$^{B}11.74^{b}$
	50	100	$^{A}9.81^{a}$	$^{B}10.89^{a}$	$^{B}11.33^{a}$
		200	$^{A}9.98^{ab}$	$^{B}10.71^{ab}$	$^{B}11.10^{ab}$
		300	$^{A}10.14^{b}$	$^{AB}10.97^{b}$	$^{B}11.19^{b}$
	80	100	$^{A}8.50^{a}$	$^{B}9.40^{a}$	$^{B}9.52^{a}$
		200	$^{A}8.16^{ab}$	$^{B}9.15^{ab}$	$^{B}9.92^{ab}$
		300	$^{A}8.51^{b}$	$^{AB}9.27^{b}$	$^{B}10.66^{b}$

Note: Lowercase (a-c) indicate statistical difference between each pressure at constant time. Uppercase (A-C) indicate statistical difference between each time at constant pressure. $P < 0.1$ (Duncan's test).

Table 2. Results of Pearson Correlation Analysis

	Temperature	Time	Pressure
Retention	0.001	0.120**	0.148**

Note: ** Significance at a 5% level

Time (β_2) was significant in the positive (+) direction at the 5% level, as was pressure (β_3). The temperature showed significance in the positive (+) direction at the level of 10%. These results represent a positive (+) effect of all independent variables on the amount of impregnation.

Using the dummy variable, a significant difference was observed in impregnation ability under the same conditions between species, with greater impregnation ability in Korean red pine than in larch. Based on this, it is presumed that pit aspiration of Korean red pine was lower than that of larch, because of the greater open-pore porosity of Korean red pine. In the future, the effects of pressure, time, and temperature on impregnation amount will be analyzed in more diverse wood samples.

The standardization coefficient was 0.024 for temperature, 0.124 for time, and 0.148 for pressure in this regression model. These results indicate that the impregnation amount was affected in the following order: pressure was greater than time, which was greater than temperature.

As pressure had the most significant influence on impregnation amount, production time can be shortened by increasing the pressure, and the temperature does not need to be considered.

Table 3. Results of Multiple Regression Analysis

$Y = \alpha_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 D_1 + \varepsilon$					
Variables	Coefficient	SE	Std Coefficient	t-stat	VIF
Intercept	0.642	0.019		34.64	
Temperature	0.001*	0	0.024	1.617	1.037
Time	0.002**	0	0.124	8.513	1.037
Pressure	0.001**	0	0.148	10.367	1
Dummy	-0.519**	0.008	-0.953	-66.587	1
Adjusted R^2			0.946		
F-value			1153.43		
p-value			< 0.001		
Note: * Significant at a 10% level; and ** significant at a 5% level					

A limitation of this study was that the maximum pressure was 300 psi, due to concerns for safety of the impregnation chamber. In the future, it is necessary to evaluate the impregnation process under ultra-high-pressure conditions after increasing the durability of the impregnation chamber.

In addition, this study investigated the effects of temperature, pressure, and time in the impregnation process of two species without pre-treatment. In previous studies, various pre-treatments such as pre-acid treatment (Yildiz *et al*. 2010), microwave treatment (Ramezanpour *et al*. 2015), and ozone treatment (Gezer and Kustas 2019) were proposed to improve the impregnation process. These pre-treatments changed the pore shape of the wood into a more open structure. It is thought that regression analysis of the impregnation process according to temperature, pressure, and time is necessary for later impregnation.

Also, this study will be developed into a wood impregnation prediction model by including and analyzing additional species.

CONCLUSIONS

1. The alkaline copper quaternary (ACQ) treatment was rapidly impregnated during the first 30 minutes.
2. Multiple regression analyses showed that the factors affecting impregnation were ranked in the order pressure, time, and temperature.
3. Pressure is the most critical parameter compared to time and temperature in wood impregnation.
4. Temperature did not significantly affect the impregnation process.

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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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