

Relationship between Chemical Retention and Velocity of Air-Coupled Ultrasonic Waves in Fire-Retardant-Treated Wood

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The ultrasonic wave velocities in wood impregnated with a fire-retardant chemical were measured *via* a non-contact method. An air-coupled ultrasonic wave was made to propagate along the radial direction of the wood. The wave velocities in the wood samples after chemical impregnation were larger than those before impregnation. With increased chemical concentration, the relative changes in the velocities increased to a maximum of 16.3%, and these velocity changes exhibited a strong correlation with the chemical retention. These findings suggest that it is possible to evaluate the uniformity distributions of chemicals in fire-retardant-treated wood *via* a non-contact and nondestructive method based on air-coupled ultrasonics.

Keywords: Fire-retardant-treated wood; Chemical retention; Air-coupled ultrasonics; Wave velocity; Nondestructive evaluation; Non-contact method; Japanese cedar

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INTRODUCTION

The revision of the Building Standard Law in 2000 made it possible to construct wooden fire-resistant structures in Japan. After this revision, a new legislation to promote wood use in public buildings was enacted in 2010. This has led to a significant increase in the number of wooden structures being built in Japan. As per standard building law, it is necessary for such structures to use fire-retardant materials in the walls and ceilings. Further, the fire-retardant-treated wood must have its fire performance accredited by the Ministry of Land, Infrastructure, Transport, and Tourism (MLIT 2011). Fire-retardant-treated wood is categorized as noncombustible, quasi-noncombustible, or fire-retardant wood *via* a fire performance test based on ISO 5660-1 (2015). However, the introduction of a cone calorimeter instrument is quite costly for wood industry factories. Generally, these facilities evaluate the fire performance of their fire-retardant-treated wood by considering the degree of chemical retention. In this context, Harada *et al.* (2003) have demonstrated that chemical retentions of 80 kg/m³ and 160 kg/m³ ensure a sufficiently safe fire performance of fire-retardant wood and quasi-noncombustible wood, respectively, *via* a cone calorimeter test. Nevertheless, some fire-retardant woods with no documented fire performance have occasionally been sold on the market. Consequently, it is important to know whether the required amount of chemical fire retardant is present in all sections of a wood sample. However, chemical retention is calculated only from the

whole weight difference before and after chemical impregnation; thus, it is difficult to determine the degree of chemical retention within an individual sample. In this regard, Kawarasaki *et al.* (2012) investigated the chemical distributions along the axial direction of long-size timbers treated with a fire retardant. They reported that there was no difference in the chemical distribution for different chemical retentions of the timber specimens. The amount of chemical retention close to the surface of the timber specimen was greater than that inside the specimen. Kamikawa (2013) suggested a water leaching method that makes it possible to determine the chemical retention of cutout wood samples. However, this procedure is not practical for in-line factory inspections. The development of nondestructive inspection systems for chemical retention in fire-retardant treated wood has thus attracted considerable attention in the context of wood industries. Recently, air-coupled ultrasonic waves have been studied for use in the quality control of wood and wood-based composites (Vun *et al.* 2008; Dahmen *et al.* 2010; Hsu *et al.* 2010; Sanabria *et al.* 2011; Sanabria *et al.* 2013; Mori *et al.* 2016). This technique makes it possible to evaluate the current state of a wood sample without contact and without destroying the wood.

In this study, the nondestructive measurement of chemical retention in fire-retardant-treated wood was experimentally investigated *via* a non-contact method based on air-coupled ultrasonics. The velocities of ultrasonic waves made to propagate through a Japanese cedar specimen treated with different chemical concentrations were measured.

EXPERIMENTAL

Materials

The sapwood of the Japanese cedar (*Cryptomeria japonica* D. Don) was used as the test material. The test specimen dimensions were 30 mm (longitudinal) by 20 mm (radial) by 35 mm (tangential), with a total of 20 test specimens. The 20 samples were divided into 5 pieces per concentration. The average air-dried density of each sample was $360 \pm 8.5 \text{ kg/m}^3$.

Treatment with Fire-Retardant Solution

A commercial fire retardant, whose main component was guanidine phosphate, was used to impregnate the wood specimens. The fire retardant was diluted with distilled water to prepare 0%, 10%, 20%, and 30% solutions. The wood specimens were placed in a vacuum desiccator at -0.09 MPa for 2 h, and subsequently they were dipped into the retardant solutions and placed in a vacuum desiccator at 0.7 MPa for 21 h. After impregnation, the specimens were air-dried for 1 month and then dried in a constant-temperature oven at 60 °C for 24 h. The chemical retention (C) was calculated using Eq. 1,

$$C = (W_a - W_b) / V \quad (1)$$

where W_a represents the specimen weight after chemical impregnation and drying at 60 °C for 24 h, W_b is the weight of the specimen dried at 60 °C for 24 h before chemical impregnation, and V represents the volume of the test specimen before chemical impregnation.

Ultrasonic Measurement

An air-coupled ultrasonic wave was made to propagate through air and the wood specimens, as shown in Fig. 1. The ultrasonic wave velocities were measured with a setup comprising of a pulser-receiver (JPN-10CKN), preamplifier, and monolithic composite transducers (14 by 20 mm-type) with a resonant frequency of 200 kHz. The distance between the transmitter and receiver transducers was set to 30 mm. The propagation directions of the ultrasonic wave corresponded to the radial direction of the wood. The propagation time at the first peak was measured with the zero-crossing method, as shown in Fig. 2. The propagation time measurement was repeated thrice, and the averaged values were used as the experimental values. The accuracy of the measurement was 1 ns. The room temperature (t °C) was measured to calculate the wave velocity in air (v_a). The ultrasonic wave velocity (v_w) was calculated *via* Eq. 2 and Eq. 3,

$$v_w = L_w / (T_1 - T_0 - (L - L_w) / v_a) \quad (2)$$

$$v_a = 331.5 + 0.61t \quad (3)$$

where L_w is the propagation distance of the given wood sample, T_1 is the propagation time with the wood sample, and T_0 is the propagation time in the absence of the sample.

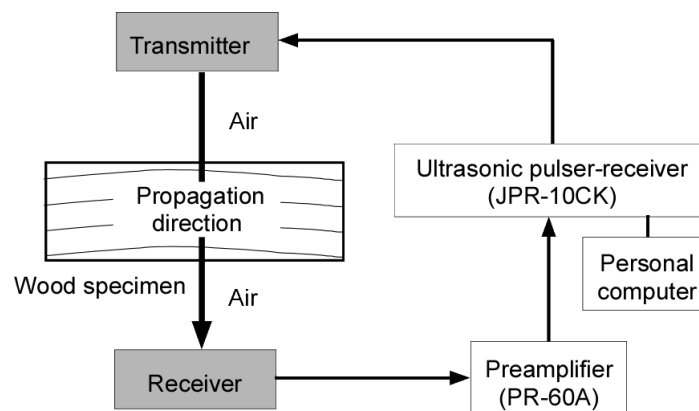


Fig. 1. Setup for ultrasonic wave-velocity measurement

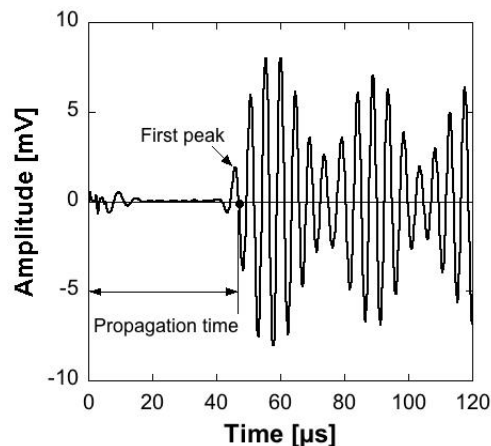


Fig. 2. Method of measuring propagation time

RESULTS AND DISCUSSION

Chemical Retention of Fire-Retardant-Treated Wood

Table 1 lists the average chemical retention and density of the samples for different chemical concentrations of the retardant. The chemical retention increased with increases in chemical concentration. The chemical retention appeared to exhibit a relationship with the density after impregnation at a 1% significance level. In this context, Chuang and Wang (2002) demonstrated that the density of a Chinese fir (*Cunninghamia lanceolata*) treated with fire-retardant chemical exhibited a significant positive relationship with the chemical retention. The maximum value of the chemical retention was 109 kg/m³. Further, Harada *et al.* (2003) demonstrated that a chemical retention of 80 kg/m³ yields a suitable fire performance for a fire-retardant wood. In this study, the test sample treated with a 30% impregnant concentration satisfied the criteria of fire-retardant wood. The sample density increased with increases in chemical concentration, and the maximum density was 444 kg/m³. The sample density after impregnation was greater than that before impregnation at a 5% significant level ($r = 0.45$). The density change rates in the 10%, 20%, and 30% concentration solutions were 1.0%, 11.7%, and 20.7%, respectively.

Table 1. Chemical Retention of Fire-Retardant in Wood Samples along with Change in Sample Density for Different Chemical Concentrations

Chemical Concentration (%)	Chemical Retention (kg/m ³)	Density (kg/m ³)	
		Before treatment	After treatment
0	0	362 (0.01)	330 (0.01)
10	32 (0.9)	352 (0.02)	355 (0.02)
20	72 (0.8)	357 (0.02)	399 (0.02)
30	109 (1.8)	368 (0.01)	444 (0.01)

Numbers in parentheses show the standard deviation.

In this section, the weight change observed in the samples after fire-retardant impregnation is discussed. In general, a water-soluble fire retardant impregnated into wood is deposited in the cell wall and lumen of the tracheid after drying. Harada *et al.* (2009) demonstrated the deposition of fire-retardant in the lumen of the tracheid of the Japanese cedar; this deposition was recorded *via* scanning electron microscopy energy-dispersive X-ray spectroscopy (SEM-EDX). A wood test specimen treated with high-chemical-concentration solution stores a large amount of fire-retardant within itself; the entries in Table 1 confirm this fact in this study. However, the density of the test sample treated with 0% chemical concentration, that is, distilled water, decreased. This result is attributed to weight decrease *via* elution of the extractives in the test sample into water. In this regard, Kamikawa has reported a weight decrease in wood samples of Japanese cedar and Japanese larch impregnated with distilled water; consequently, the weight decrease due to the extractives must be accounted for in the calculations (2013).

Relationship between Chemical Retention and Ultrasonic Wave Velocities

Figure 3 shows an example of the received waveform before and after chemical impregnation with 10% chemical concentration. The amplitude of the received waveform is decreased after chemical impregnation. However, the amplitude decrease due to chemical impregnation was not common to all the samples impregnated with different

chemical concentrations. Table 2 lists the average ultrasonic wave velocities before and after fire-retardant impregnation with different chemical concentrations. The average velocities before and after fire-retardant impregnation ranged from 2137 m/s to 2438 m/s and 2250 m/s to 2656 m/s, respectively. Figure 4 shows the relationships between the wave velocities before and after chemical impregnation. The velocity after impregnation (v_{wa}) was greater than that before impregnation (v_{wb}) at a 1% significance level ($r = 0.78$). The percentage change in the velocity can be expressed as the ratio of the relative change in velocity after impregnation to the velocity before impregnation, as shown in Eq. 4.

$$\text{Percentage change in } = (v_{wa} - v_{wb}) / v_{wb} \times 100 \tag{4}$$

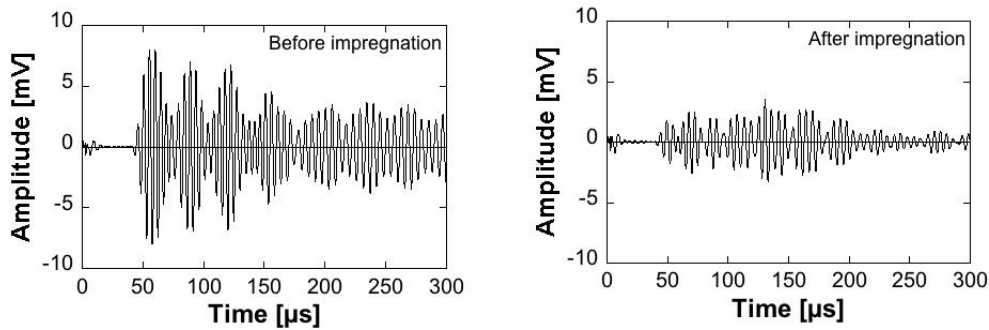


Fig. 3. Received waveforms before and after chemical impregnation

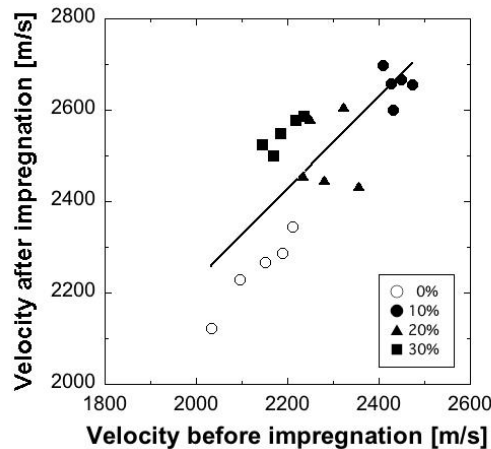


Fig. 4. Relationships between wave velocities before and after chemical impregnation. The legend indicates the concentration associated with each data point.

Table 2. Ultrasonic Wave Velocity Before and After Impregnation in Wood for Different Chemical Concentrations

Chemical Concentration (%)	Velocity (m/s)		Percentage Changes in Velocity (%)
	Before treatment	After treatment	
0	2137 (72)	2250 (82)	5.3
10	2438 (24)	2656 (35)	8.9
20	2288 (50)	2504 (81)	9.5
30	2190 (36)	2548 (37)	16.3

Numbers in parentheses show the standard deviation.

Figure 5 shows the variation in the velocity change due to chemical retention. The velocity change was 8.9% for a chemical concentration of 10%, and the velocity change increased with increases in chemical retention. The maximum velocity change (16.3%) was observed for a retardant concentration of 30%. Figure 6 shows the relationship between the chemical retention and the percentage change in ultrasonic wave velocity. The percentage change in velocity exhibited a positive correlation with the chemical retention ($r = 0.94$).

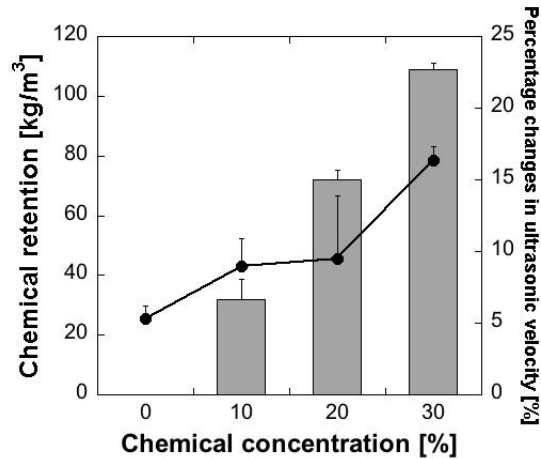


Fig. 5. Chemical retention and percentage change in ultrasonic velocity for different chemical concentrations

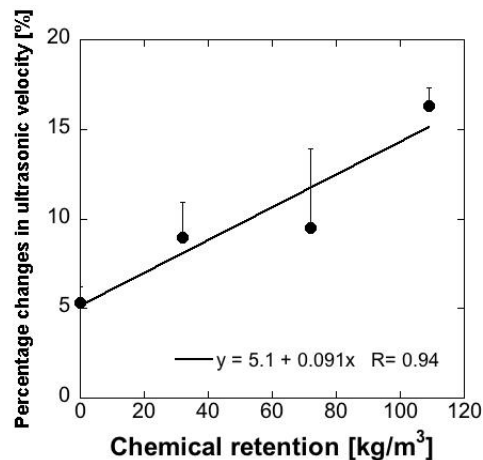


Fig. 6. Relationship between chemical retention and percentage change in ultrasonic velocity

The velocity of an ultrasonic wave propagating through a material is generally affected by the material density. Materials with high densities correspond to high propagation velocities. In the case of wood and wood products, several researchers have examined the effect of wood density on the wave velocity (Bucur and Chivers 1991; Mishiro 1996; Oliveira and Sales 2006; Hasegawa *et al.* 2011). In this context, Mishiro (1996) reported that the velocities along the radial and tangential directions increased with increases in density; however, the wave velocities were found to be independent of the density upon eliminating the lightest (balsa (*Ochroma pyramidale*)) and heaviest (Lignum vitae (*Guaiacum sanctum*)) woods. Hasegawa *et al.* (2011) reported that the velocities in the radial and tangential directions exhibited almost no correlation with

density for Japanese cedar and Japanese cypress. Thus far, no studies have reported any correlation between the velocities in the radial and tangential directions and the wood density. However, Vun *et al.* (2008) measured the ultrasonic wave velocity in the direction perpendicular to the surface of an oriented strand board (OSB) treated with different resin concentrations. These authors demonstrated that the ultrasonic wave velocity increased nonlinearly with increases in the apparent density of the OSB. In this study, the apparent density of the test specimens increased due to the chemical impregnation, as can be inferred from Table 1. As a result, the wave velocity in the test specimen treated with a high-concentration solution greatly increased as a result of the chemical impregnation.

In this study, the relationships between the chemical retention and percentage changes in velocity were experimentally confirmed for the first time, to the best of the authors' knowledge. These results indicate that the ultrasonic wave velocity through fire-retardant-treated wood can be utilized to evaluate the chemical retention in the wood. In the future, we plan to investigate the application of an ultrasonic technique to nondestructive prediction of the distribution of fire-retardant in a given wood sample, as it is necessary to evaluate the chemical distribution in actual-size specimens for practical applications; in the current study, only small wood specimens were used to measure the ultrasonic wave velocity. In addition, we plan to conduct a cone calorimeter test to validate nondestructive measurements of chemical retention *via* air-coupled ultrasonics.

CONCLUSIONS

1. The ultrasonic wave velocity after impregnation was greater than that before impregnation at a 1% significance level ($r = 0.78$).
2. The ultrasonic wave velocity change increased with an increase in chemical retention. The maximum velocity change (16.3%) was observed for a retardant concentration of 30%.
3. The percent changes in ultrasonic wave velocity exhibited a positive correlation with the chemical retention.

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