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Weatherability and combustibility of fire-retardant-impregnated wood after accelerated weathering tests

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Abstract The weatherability and combustibility of fire-retardant-impregnated wood during accelerated weathering for up to 2000 h were evaluated. The ability of coating to retain fire-retardant chemicals against leaching was also examined using four coating systems (water-borne or solvent-borne, pigmented or clear, film-forming or penetrating). Furthermore, the distribution of fire retardants in the specimens was observed by scanning electron microscopy in combination with energy dispersive analysis of X-rays (SEM-EDX). The fire performance of the specimens during weathering depended on the chemical retention, and was maintained at a quasi-noncombustible material level if the chemical retention was above 150 kg/m³. The maximum duration of accelerated weathering to keep this retention was 250 h for the uncoated specimens, but increased to over 1000 h for pigmented coatings. SEM-EDX revealed that the fire retardants were accumulated in the cell lumina throughout the wood tissues. However, in the uncoated samples, the leaching of fire retardants occurred for surfaces exposed to light and water, and was observed down to a depth of ~150 μm after 500 h. The leaching depth extended throughout the specimen after 1000 h. In contrast, the fire retardants still remained in samples finished with a solvent-borne pigmented penetrating coating even after 1000 h due to their relatively high chemical retention.

Key words Fire-retardant wood · Cone calorimeter · Accelerated weathering test · SEM · EDX

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Introduction

The use of wood in exterior applications has increased since 2000, when revisions in the Building Standard Law of Japan started to permit the use of wooden exterior walls if the relevant fire performance criteria were met. It is well known that the fire performance of fire-retardant-treated (FRT) wood depends on the amount of fire-retardant chemical (FRC) impregnated into the wood. In the case of phosphorous and nitrogen chemicals, the required amount of chemicals is 70–100 kg/m³ to meet the criteria for fire retardant material and 150–200 kg/m³ for quasi-noncombustible material, according to the Building Standard Law of Japan.¹ However, because the FRC for wood is water soluble, there is a concern that the fire performance of FRT wood is degraded in exterior applications.

The leaching mechanism of FRC from FRT wood due to weathering has not been fully elucidated, but some important aspects of the phenomenon are explained as follows. The photodegradation and defiberization of the surface wood cells are induced by exposure to ultraviolet (UV) light. These degradation processes are enhanced by the presence of water. The water absorbed during rainfall penetrates into wood, and this causes slow diffusion of the FRCs toward the surface with resultant loss of the chemicals. However, there have been few studies that have reported on the rate of FRC leaching and its influence on the fire performance of FRT wood. There is also still little information on the distribution and diffusion of FRC during weathering and how to prevent it.

The weatherability of FRT wood has already been discussed in Forest Products Laboratory (FPL) reports^{2–4} and Östman's reviews.^{5,6} According to the FPL reports,^{2,3} which describe natural weathering tests that were conducted for up to 10 years, some of the treatments such as the impregnation of dicyandiamide–phosphoric acid–formaldehyde showed acceptable performance. Fire tests for roof coverings (Burning brand tests⁷) and vertical flame spread tests (modified Schlyter tests³) were also conducted. However, the values for chemical retention after weather-

ing were not shown, and it is not clear whether their results correspond to the fire performance evaluated by the cone calorimeter test currently being used in Japan.

The average heat release rates for the leached FRT wood were measured by heat release calorimeter,⁴ but these specimens were leached by placing them in distilled water. More recently, Östman and Tsantaridis⁶ conducted cone calorimeter tests of FRT wood after leaching by accelerated weathering (ASTM D2898-94)⁸ and natural weathering, and concluded that the fire performance of FRT wood could be maintained if the retention levels were high enough. They also reported that paint systems contribute considerably to maintaining the fire performance of FRT wood in exterior applications. However, these results cannot simply be adopted in Japan because of the differences in the evaluation system of fire performance between countries. In addition, the distribution of the FRC during weathering was not discussed.

In this article, the weatherability of FRT woods was evaluated using a xenon weather meter and their combustibility was rated by cone calorimeter test to study quantitatively the rate of FRC leaching and the influence of FRC loss on the fire performance. The effect of coating to prevent leaching was also examined using several types of coating systems. Furthermore, the distribution of FRC in the specimens with and without coating was monitored using scanning electron microscopy in combination with energy-dispersive X-ray analysis (SEM-EDX) to examine the leaching mechanism of FRC and the protective effect of coating.

Experimental

Wood specimens

Sugi (*Cryptomeria japonica* D. Don) sapwood specimens measuring 100 mm (tangential) × 120 mm (longitudinal) × 8 mm (radial) were impregnated with FRC (polyphosphatic carbamate), and were then finished with one of four coating systems as will be described. After accelerated weathering

tests, the samples were each cut into two pieces: a large one for the cone calorimeter test, and a smaller one for the observations of FRC distribution and coating systems by SEM-EDX.

Fire-retardant treatment

The FRC used to treat the wood specimen was the commercially available Non-nen OK-201 (Marubishi Oil Chemical). The concentration of the undiluted solution was 50%. The main component was polyphosphatic carbamate and the degree of polymerization was 5–10. The chemical compound was $\text{NH}_2\text{CO}[\text{OPO}(\text{ONH}_2)]_n\text{-OCNH}_2$. The fire retardant was diluted by water to prepare a 25% solution.

The processing procedure was as follows. The specimens were immersed in the fire-retardant solution and placed in a vacuum at 25 torr (3333 Pa) for 30 min, subjected to pressurized air [10 kgf/cm^2 (0.98 MPa)] for 2 h, and then placed in a vacuum again for 30 min. After impregnation, the specimens were removed from the chemical feeder and dried under atmospheric conditions for 1 week and then at 60°C for 10 days until the specimen mass stayed constant. The dry chemical retention was approximately 220 kg/m^3 . This amount was calculated from the weight of treating solution that each specimen had absorbed during the treating process and the concentration of the solution.

Coating

After the impregnation treatment, the front, back, and lateral surfaces of the specimens were finished with one of four coating systems and their end grains were sealed with rubber-based resin. The applied coating systems were (A) water-borne film-forming opaque coating, (B) solvent-borne pigmented penetrating coating, (C) solvent-borne thin-film-forming pigmented coating, and (D) solvent-borne polyurethane fire-retardant film-forming clear coating, as shown in Table 1. For comparison, specimens with no impregnation treatment and no coating were also prepared. Five specimens were prepared for each combination of treatment and coating.

Table 1. Overview of coating systems used

Coating system	Description	Primer			Topcoat		
		Number of layers	Mean coating weight ^a (g/m ²)	Solid content (%)	Number of layers	Mean coating weight ^a (g/m ²)	Solid content (%)
A Water-borne acrylic emulsion paint	Film-forming; opaque; brown	1	84.3	10.3	2	96.7 166.4	46.6
B Solvent-borne alkyd stain 1	Penetrating; semitransparent; brown	0	–	–	2	118.1 89.3	28.9
C Solvent-borne alkyd stain 2	Thin-film-forming; penetrating; semitransparent; brown	0	–	–	2	116.3 93.9	32.9
D Solvent-borne polyurethane clear coating	Film-forming; transparent	1	123.8	25.9	1	103.9	20.0

^a Measured immediately after brush coating in grams wet paint per unit area

Accelerated weathering test

The weatherability of specimens was assessed in an accelerated weathering chamber (Atras Ci4000) according to JIS K5600-7-7 (xenon lamp method)⁹ for up to 2000 h. The accelerated weathering regime involved continuous exposure to xenon arc UV radiation (0.5 W/m^2 at $340 \text{ nm} = 550 \text{ W/m}^2$ for $290\text{--}800 \text{ nm}$), and 18 min of deionized water spray every 2 h with a black panel temperature of $65^\circ \pm 2^\circ\text{C}$ and a chamber temperature of $40^\circ \pm 2^\circ\text{C}$. Mass loss, retention of chemicals, color change, and water repellency (WR) were evaluated. Specimen mass was measured before and after 250, 500, 750, 1500, and 2000 h of weathering. The retention of chemicals was calculated according to Eq. 1, after the specimens were dried to a constant mass in a dry oven at 60°C . In this study, all of the mass lost during the accelerated weathering test was regarded as the fire-retardant decrease and the leaching of water-soluble wood extractives was neglected assuming that most of them had been leached out during the fire-retardant impregnation process.

$$R_h = R_0 - (W_0 - W_h)/V \quad (1)$$

where R_h is the retention of chemicals (kg/m^3) after h hours of weathering, R_0 is the initial retention of chemicals (kg/m^3), W_0 is the initial specimen mass (kg), W_h is the specimen mass (kg) after h hours of weathering, and V is the specimen volume (m^3).

Color change and water repellent index were measured before and after 500, 1000, 1500, and 2000 h of weathering. A colorimeter (Nihon Denshoku Kogyo NF333) was used to quantify color change at the surface of the specimen as a result of weathering. Color is expressed using the CIE Lab (L^* , a^* , b^*) system. Color change (ΔE_{ab}^*) was calculated according to Eq. 2.

$$\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2)$$

Water repellency (WR)¹⁰ is expressed as a percentage and was determined according to Eq. 3.

$$\text{WR} = [1 - (W_2 - W)/(W_1 - W)] \times 100 \quad (3)$$

where W is the specimen mass before the test, W_1 is the specimen mass with approximately 1 g of water dropped with a syringe on the surface, W_2 is the specimen mass after the dropped water was wiped off with soft paper 1 min later.

Fire test

The fire performance of the specimens was evaluated by a cone calorimeter¹¹ (Toyo Seiki Cone3a). Specimens were prepared with dimensions of 100 mm (tangential) \times 100 mm (longitudinal) \times 8 mm (radial), and were placed horizontally under a cone heater with a heat flux of 50 kW/m^2 . The heating time was 10 min, the same time used for the quasi-noncombustible material test. A stainless steel cover with an opening of 0.0088 m^2 on the upper part was attached. The data were recorded by a computer every 2 s, and the

total heat release for 5 and 10 min (THR_5 , THR_{10}) was evaluated.

Cone calorimeter tests were conducted for coated specimens before and after 500, 1000, 1500, and 2000 h of weathering. For the uncoated specimens, the tests were done before and after 250, 500, 1000, and 2000 h of weathering. Specimens with the largest mass loss for the same treatment and weathering condition were selected for the cone calorimeter tests.

SEM-EDX technique

A sledge microtome was used to reveal the transverse surface of specimens measuring $10 \times 10 \times 8 \text{ mm}$ obtained from unweathered and weathered samples. They were coated with carbon and examined using a scanning electron microscope (JEOL JSM-5600LV) equipped with an energy-dispersive X-ray detector (JEOL JED-2140). This microscope was used to examine and map the distribution of phosphorus in FRT wood samples. The microscope used an accelerating voltage of 15 kV; illuminating current of 1.5 nA; working distance of 20 mm; takeoff and tilt angles of 30° and 0° , respectively, and dead and dwell (digital map) times of 15%–20% and 0.2 ms/point, respectively. The elemental peaks detected were P-K α using a window width of 1.96–2.07 keV. Back-scattered electron images were used in preference to secondary electron images because they gave better contrast between polyphosphoric carbamate and wood.

Results and discussion

Chemical retention

Figure 1 shows the average retentions of FRC for up to 2000 h of accelerated weathering. In the case of specimens without coating, the FRC retention decreased steeply from the beginning. The initial chemical retention of 220 kg/m^3 decreased to 76 kg/m^3 at 500 h, 27 kg/m^3 at 1000 h, and 11 kg/m^3 at 2000 h. On the other hand, specimens with coatings had chemical retention of $175\text{--}191 \text{ kg/m}^3$ after 750 h of weathering. However, differences could be seen in leach resistance at 1000 h and later depending on the type of coating. The chemical retention of type B was 150 kg/m^3 , whereas that of types A and C was 80 kg/m^3 and that of type D decreased to the same level of retention as the uncoated specimens (13 kg/m^3) after 2000 h of weathering.

The higher retention values for the coated specimens compared with the uncoated specimens indicate that the leaching of FRC can be reduced to a considerable extent by coating. Our results are in accordance with those of Östman and Tsantaridis⁶ who suggested the protective effect of coating in preventing FRC leaching.

It was also found in our study that the protective effect of coating can be enhanced by the presence of pigments in the coating systems. Figure 1 shows that the retention values for the pigmented coatings A, B, and C were greater than that of the clear coating D.

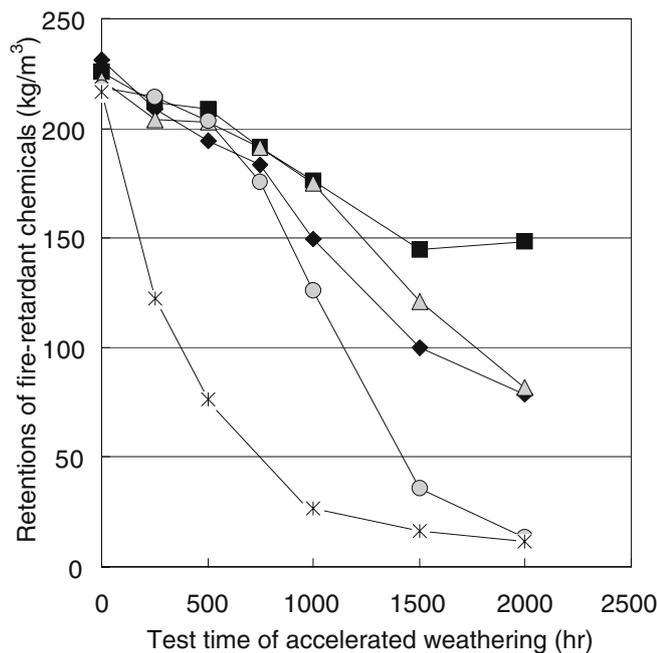


Fig. 1. Retentions of fire-retardant chemicals for up to 2000 h of accelerated weathering. *Diamonds*, water-borne film-forming opaque coating A (see Table 1); *squares*, solvent-borne pigmented penetrating coating B; *triangles*, solvent-borne thin-film-forming pigmented coating C; *circles*, solvent-borne polyurethane fire-retardant film-forming clear coating D; *stars*, no coating

The difference in coating weight is also considered to be a contributing factor. Mean dry coating weights of coating systems A, B, C, and D were calculated to be 123, 60, 69, and 21 g/m², respectively, based on their wet weights and solid contents in Table 1. Lower coating weight may be one reason for more leaching but it is generally known that the weatherability of clear coating is inferior to pigmented coating. In this study, the performance of coatings after accelerated weathering tests as mentioned later supported this notion and it is considered that clear coating D allowed UV penetration more easily and deterioration of the wood-coating interface occurred more quickly than with the pigmented coatings.

It was interesting that among the pigmented coatings used here, the penetrating type B showed better retention values than the film-forming types A and C. As discussed below, SEM observation revealed that coating B penetrated into wood surface cells to generate a protective zone beneath the surface, instead of forming a film on it. The difference in the protection mechanism between the coatings of the highest and the lowest retentions is discussed below in terms of results from SEM-EDX analysis.

Performance of coatings on FRT wood

It is important to evaluate the performance of coating systems on FRT wood because FRC in wood may negatively affect it. Thus, changes in WR and changes in color were monitored during accelerated weathering, as shown in Figs. 2 and 3, respectively.

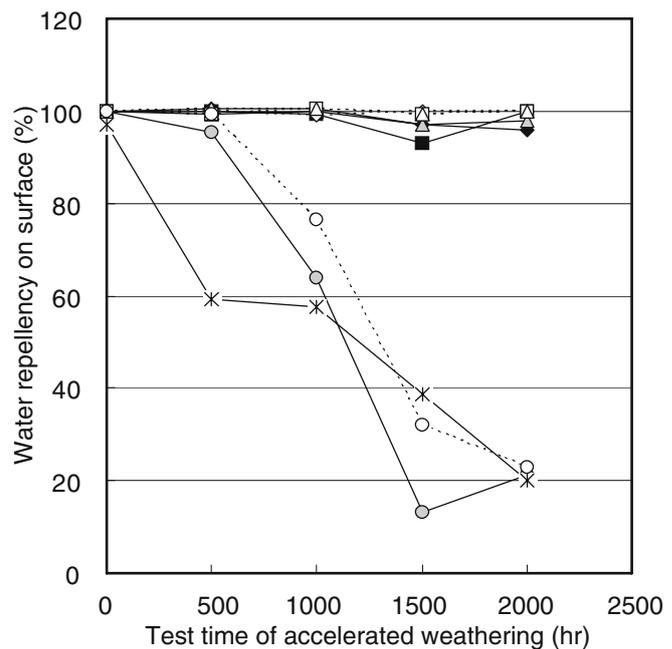


Fig. 2. Water repellency on surface for up to 2000 h of accelerated weathering. *Diamonds*, coating A; *squares*, coating B; *triangles*, coating C; *circles*, coating D; *stars*, no coating; *filled symbols*, fire-retardant-impregnated samples; *open symbols*, fire-retardant-free samples

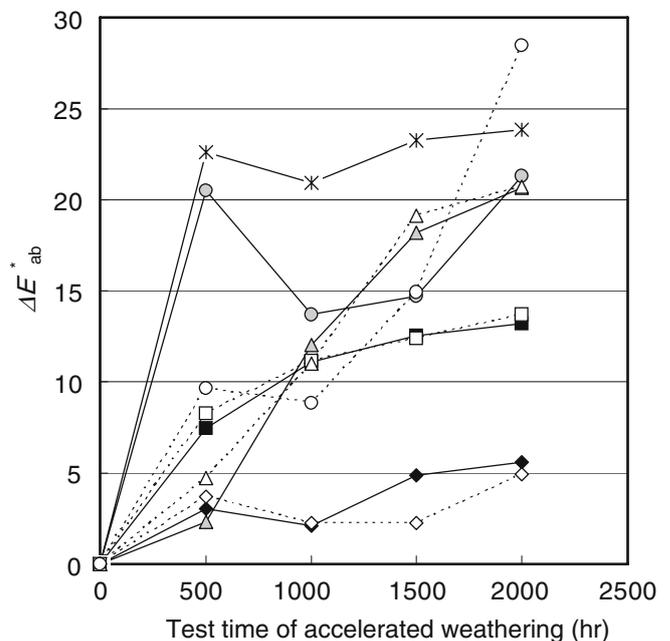


Fig. 3. Color change on surface for up to 2000 h of accelerated weathering. Symbols correspond to those used in Fig. 2

It can be seen in Fig. 2 that the specimens with the pigmented coatings A, B, and C showed much smaller changes in WR than specimens coated with the clear coating D, which started to decrease in WR after 500 h and dropped to almost the same level as for the uncoated specimens at 1000 h. This result agrees with the results for FRC reten-

tion, which also demonstrated that the protective effect of coating was enhanced by the presence of pigments.

However, it appeared that such effects of coatings could be degraded by the presence of FRC in wood. Figure 2 shows that WR of the coated specimens was slightly lower for the FRT wood than for FRC-free wood. This tendency was most obvious for the specimens treated with coating D, suggesting that the negative impact of FRC on the coating performance may have become more significant for the coatings with relatively low performance.

Figure 3 shows color differences (ΔE_{ab}^*) on the surface during the accelerated weathering. The pigmented coatings A, B, and C tended to show smaller ΔE_{ab}^* than the clear coating D, particularly for the specimens treated with coating A, which showed ΔE_{ab}^* values of less than 6 throughout the weathering test. However, ΔE_{ab}^* for specimens treated with coating C became as large as the value for specimens treated with coating D with increasing weathering time. These results are in accordance with those mentioned above, which suggested that pigments were effective in preventing degradation.

On the other hand, it was not clear whether the presence of FRC enhanced the color change, but the large difference observed at 500 h between samples with and without FRC for specimens treated with coating D may suggest it. Thus, attention should be paid to such negative effects of FRC on the coating performance, although this depends on both the type of FRC and the coating system.

Fire performance

Figure 4 illustrates the relationships between THR_{10} and the duration of accelerated weathering. THR_{10} of fire-

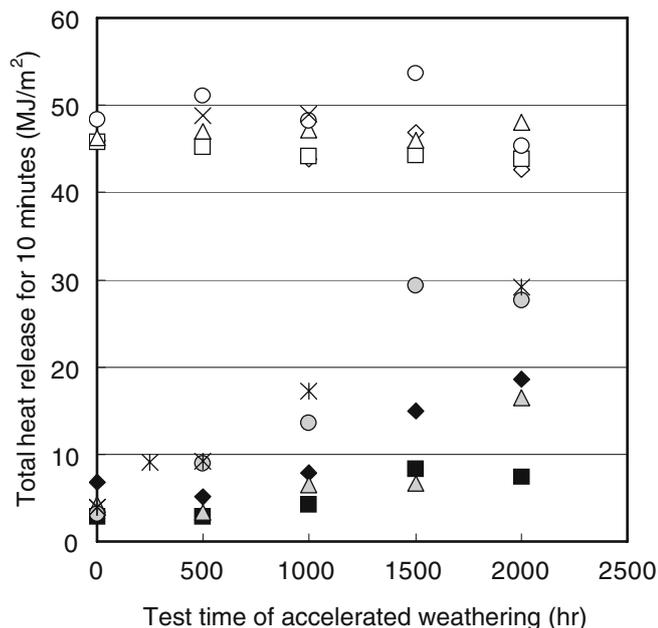


Fig. 4. Total heat release for 10-min cone calorimeter tests for up to 2000 h of accelerated weathering. Cross, fire-retardant-free sample with no coating; other symbols correspond to those used in Fig. 2

retardant-free specimen varied from 42 to 54 MJ/m² and this depended on the mass difference between wood specimens rather than the effect of coatings. THR_{10} of FRT woods before weathering was less than 8 MJ/m², which is the value required for quasi-noncombustible materials in the Building Standard Law of Japan.

For specimens treated with coating D and those without coating, THR_{10} exceeded 8 MJ/m² at 500 h and went up to 14–17 MJ/m² at 1000 h and 27–29 MJ/m² at 2000 h, respectively. Their chemical retention was 11–13 kg/m³, which is 5% of the initial retention. However, the THR_{10} values were 60% of the FRC-free wood specimen, and it is said that a low retention of FRC may have a certain degree of effect on fire performance.

THR_{10} of specimens coated with A, B, or C was less than 8 MJ/m² for up to 1000 h. For up to 2000 h, THR_{10} was 7.4 MJ/m² for specimens coated with B and 17–19 MJ/m² for specimens coated with A or C.

Figure 5 shows the relationships between the retentions of FRC in wood and their fire performance. As a previous study¹ reported, THR_{10} correlated with the retention of FRC in wood. However, after accelerated weathering tests, THR_{10} of specimens treated with coating D was higher than other coated specimens with the same amount of chemical retention. Coating system D deteriorated more quickly than the others and fire-retardant chemicals were partially leached out through checking as observed in SEM-EDX analysis. Therefore, THR_{10} of specimens treated with coating D was high for its chemical retention. In this study, the fire performance of FRT wood could be maintained at a quasi-noncombustible material level if WR was maintained near 100% and the retention of FRC was kept at more than 150 kg/m³ even after leaching. Without coating, more than 60 kg/m³ of FRC was retained at 500 h, THR_{10}

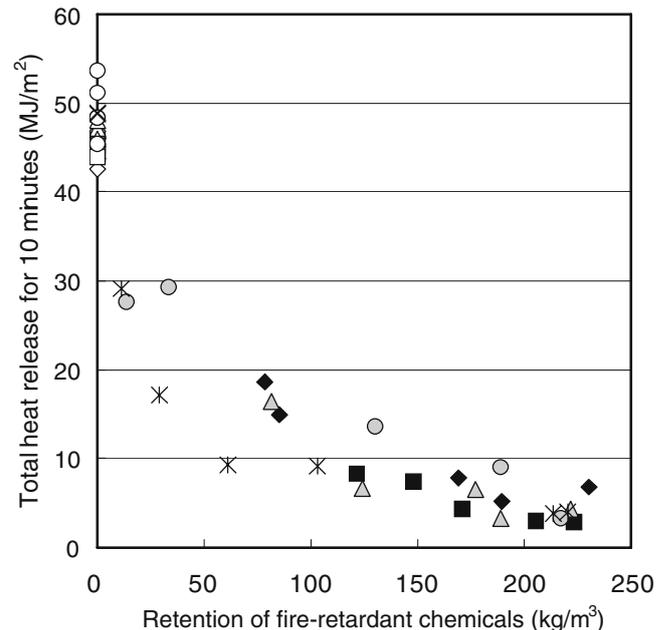


Fig. 5. Relationships between fire-retardant chemical retention and total heat release for 10-min cone calorimeter tests. Symbols correspond to those used in Fig. 4

was 9.3 MJ/m^2 , but THR_5 was 4.2 MJ/m^2 and the fire performance was equivalent to the fire-retardant material level.

There were no clear correlations between fire performance, WR, and color change. However, WR can become one of the evaluation indices used for maintaining fire-retardant-impregnated and surface-coated wood, because decreases in WR reflect the deterioration of the wood-coating interface that accelerates the leaching of FRC.

FRC distribution and protective effect of coating system

Figures 6–11 show scanning backscattered electron and EDX mapping images from sections of the unweathered and weathered FRT wood specimens with and without coating. Numerous white deposits can be clearly seen in unweathered specimens with no coating (Fig. 6A). These deposits are accumulated in the lumina of tracheids. EDX

Fig. 6A, B. Scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX) color mapping of a cross section of fire-retardant-treated (FRT) sugi sapwood uncoated and unweathered: **A** backscattered electron image; **B** P-K α X-ray image. The warmth of colors in the map represents the intensity of X-rays, with white indicating a high intensity, and therefore a high concentration of phosphorus

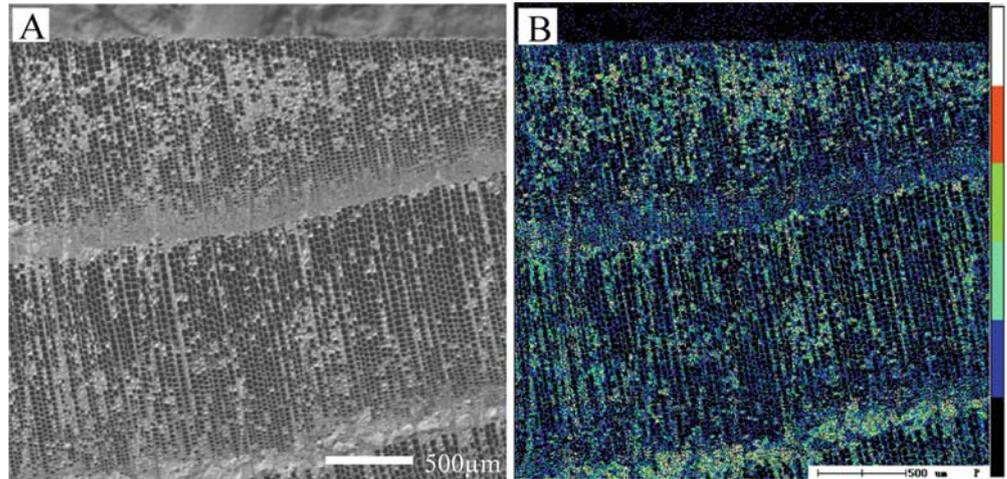


Fig. 7A, B. SEM-EDX color mapping of a cross section of FRT sugi sapwood uncoated and exposed to 500 h of accelerated weathering: **A** backscattered electron image; **B** P-K α X-ray image. Arrows mark erosion

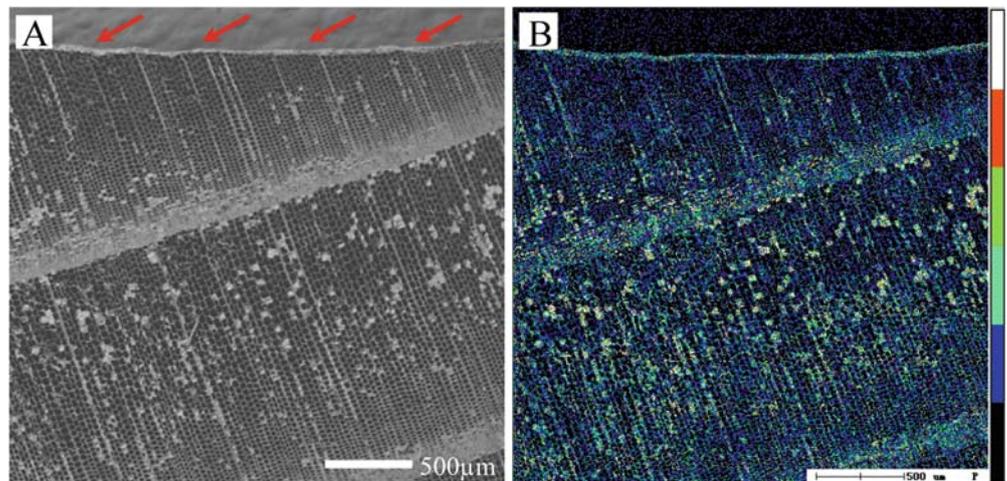


Fig. 8A, B. SEM-EDX color mapping of a cross section of FRT sugi sapwood uncoated and exposed to 1000 h of accelerated weathering: **A** backscattered electron image; **B** P-K α X-ray image

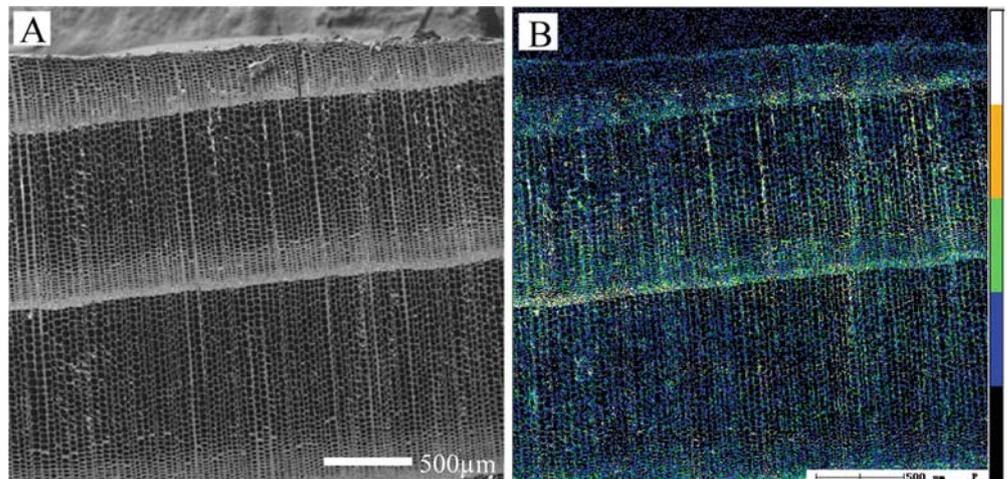


Fig. 9A, B. SEM-EDX color mapping of a cross section of FRT sugi sapwood treated with coating D and exposed to 500 h of accelerated weathering: **A** backscattered electron image; **B** P-K α X-ray image. *Arrows* mark checking

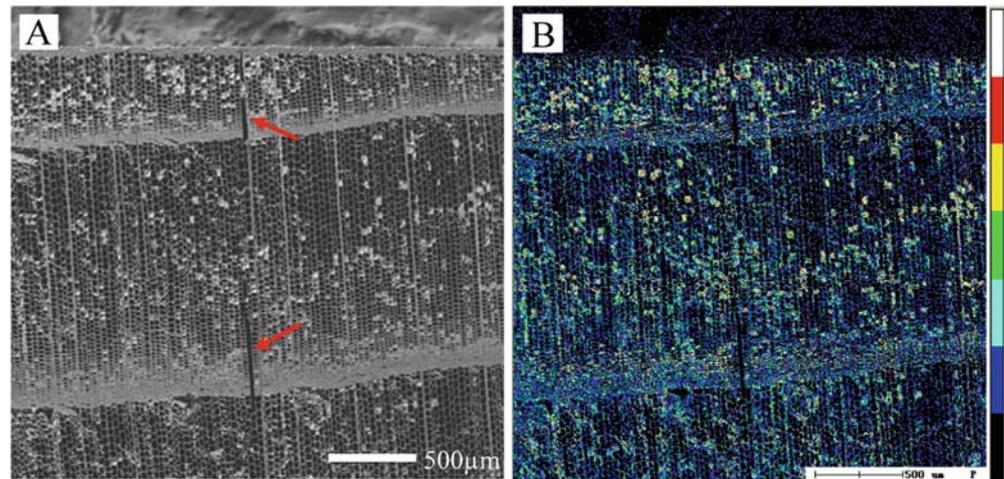


Fig. 10A, B. SEM-EDX color mapping of a cross section of FRT sugi sapwood treated with coating D and exposed to 1000 h of accelerated weathering: **A** backscattered electron image; **B** P-K α X-ray image. *Circled areas* show where the fire-retardant chemical leached out from the checking

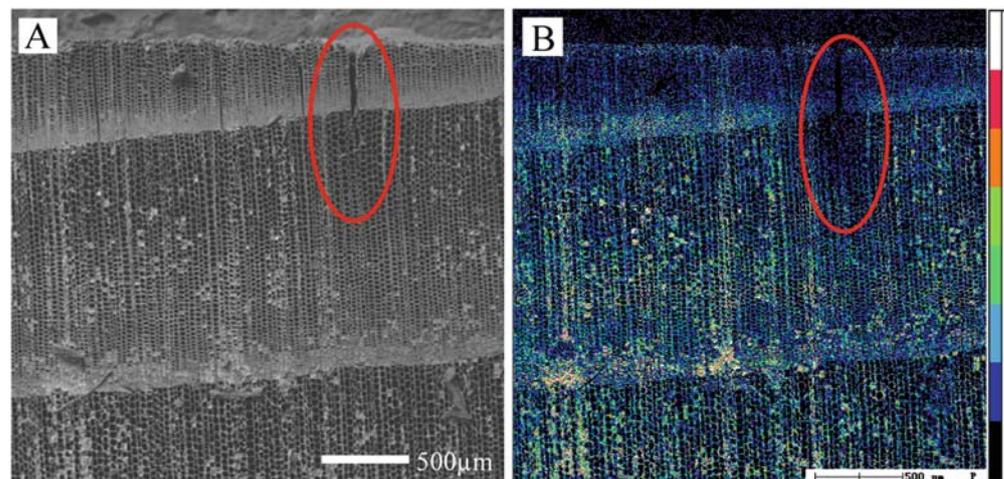
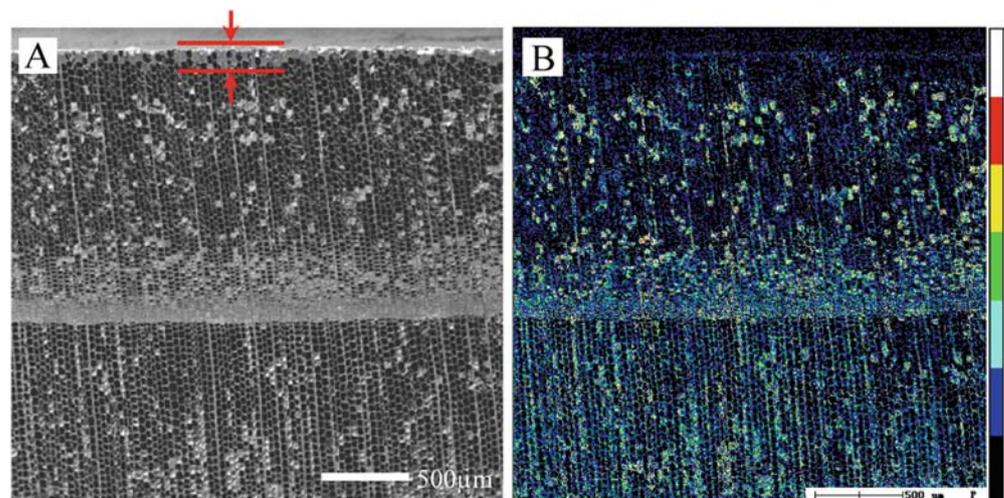


Fig. 11A, B. SEM-EDX color mapping of a cross section of FRT sugi sapwood treated with coating B and exposed to 1000 h of accelerated weathering: **A** backscattered electron image; **B** P-K α X-ray image. *Arrowed areas* show where coating B remains in the surface tracheids



mapping confirmed that the deposits contained phosphorus (Fig. 6B). The uncoated samples exposed to accelerated weathering for 500 h underwent surface erosion (Fig. 7A). Leaching of phosphorus deposits from the lumina of tracheids was also observed down to a depth of $\sim 150 \mu\text{m}$ from the surface. The leaching depth was then broadly extended

throughout the specimen after 1000 h of accelerated weathering (Fig. 8).

Unlike wood preservatives, fire-retardants such as boric acid, borax, and ammonium phosphate are not fixative types because they are basically used to treat wood for indoor use. The polyphosphatic carbamate used in this

study was also easily leached out (Figs. 7, 8) by accelerated weathering.

The effect of coating to prevent the leaching of FRC was examined by comparing coatings D and B, which show the lowest and the highest retention values in Fig. 1, respectively. Figure 9 represents an FRT wood specimen coated with the clear, film-forming coating D and exposed to accelerated weathering for 500 h. A coating layer remained on the surface, but checking could be seen (Fig. 9A), which may explain why the deposits were still present in the lumina of tracheids (Fig. 9B).

The 1000-h exposure to accelerated weathering, however, made the clear coating D exfoliate and caused surface erosion. The FRC was leached out from the weathered surface, especially through checks developed from the surface (Fig. 10). As mentioned earlier, the protective effect of clear coating D may have decreased more rapidly compared with the pigmented coatings due to UV degradation of the wood-coating interface.

In contrast, the pigmented, penetrating coating B had the greatest ability to keep the FRC within the wood (Fig. 11). In Fig. 11, it can be seen that coating B penetrated into the tracheid lumina beneath the surface. This protective zone still remained even after 1000 h of accelerated weathering (Fig. 11A). This may explain why the FRC deposits were also present at 1000 h (Fig. 11B).

The distribution of the FRC was similar to that of the unweathered control (Fig. 6). The results may account for why the total heat release of this sample was as low as the control sample (0 h, no coating).

Conclusions

The fire performance of FRT wood during accelerated weathering depended on the retention of FRC against leaching. Pigmented coatings for surface protection showed better retentions than the clear coating, presumably because of their ability to decrease UV penetration into the wood-coating interface. The best retention achieved by pigmented and penetrating coatings shows the possibility of keeping the fire performance required for quasi-noncombustible material, even after 2000 h of accelerated weathering. SEM-EDX revealed that leaching of FRC occurred on uncoated surfaces to a depth of ~150 µm after 500 h and extended throughout the specimen after 1000 h. In contrast, leaching was not significant for coated surfaces if the coating substances remained as a barrier. These results indicate that

the proper combination of FRC and coating may enable FRT wood to be used in exterior applications. Further research will compare the fire performance of FRT woods exposed to natural and artificial weathering.

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