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Properties of Colloidal Silica-Fixed and Propionylated Wood Composite (II): Flame Resistance and Other Properties of the Composites

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Abstract: The flame resistance, color change, strength, and decay resistance of colloidal silica-fixed (CSW), propionylated only, and propionylated dual-treated wood (CSPW) composites were evaluated. The oxygen indexes of the CSPW composites were similar to the CSW composites but much higher than those of untreated woods and the propionylated woods. The oxygen indexes increased with an increase in the weight percent gain of the colloidal silica (WPG_{csi}) in the composites, showing an effective reduction in the flammability by the fixation of colloidal silica. The CSPW composites showed little or no difference in the modulus of elasticity and modulus of rupture compared with the untreated woods, indicating little or no significant reduction in strength properties of the wood specimens. The color difference of the wood specimens before and after treatment changed slightly. Minimal weight losses

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of the CSPW composites occurred upon fungal attack by *T. versicolor* and *F. palustris*, showing good decay resistance by propionylation of the composites.

Keywords: Colloidal silica, flame resistance, color change, strength property, decay resistance

INTRODUCTION

Wood has served man since he appeared on Earth, and has decisively contributed to his survival and to the development of civilization. Further, wood continues to be the raw material for many products even in modern times, although other competitive materials (plastics, cement, and metals) are available. Lately, wood as renewable natural raw material is receiving more attention because of concerns of other resources. In order to make wood more competitive with plastics, metals, and other materials, the chemical modification of wood to enhance its properties of dimensional instability, flame resistance, decay resistance, and so on is being extensively studied.

Acetylation of wood using acetic anhydride can endow the wood with good dimensional stability and decay resistance.^[1–3] It was also found that propionylation of wood using propionic anhydride, a chemical modification method similar to acetylation, could give wood good dimensional instability.^[4] Propionylation should also endow wood with good decay resistance.

The wood–inorganic substance composites using the water glass-boron compound system has been used to enhance wood properties, particularly flame resistance and decay resistance.^[5–8]

Our previous study^[9] established that colloidal silica-fixed and propionylated dual-treated wood (CSPW) composites and propionylated only-treated wood showed good dimensional stability. More study is needed for a better understanding of the properties of the propionylated wood-inorganic substance composites. In this article we report on the flame resistance, decay resistance, color change, and strength properties of colloidal silica-fixed and propionylated dual-treated wood (CSPW) composites.

MATERIALS AND METHODS

Wood Specimens

Sapwoods of sugi (*Cryptomeria japonica* D. Don) were used for producing the wood-based composites. The sizes of specimens were 30 (R) × 30 (T) × 5 (L) mm for the color change test, 3.0 (R) × 6.5 (T) × 10 (L) mm for the flame resistance test, 20 (R) × 20 (T) × 10 (L) mm for the decay resistance test, and 20 (R) × 20 (T) × 320 (L) mm for strength property tests. The specimens, with the exception of those for strength property tests, were

extracted with ethanol-benzene (1:2, vol) for 12 h, and with acetone for another 12 h using a Soxhlet apparatus. The specimens used for strength property tests were initially immersed in ethanol-benzene (1:2, vol) for 7 days and in boiling water for another 12 h. All of the extracted specimens were dried at 105°C for 24–48 h, and the oven-dried weights and sizes of specimens were measured. Nine specimens were used for each condition of decay resistance test and five specimens were used for each condition of other tests.

Chemical Agents

The chemical agents were the same as mentioned previously.^[9]

Preparation of the Colloidal Silica Fixed-Wood (CSW) Composites

The CSW composites were prepared as described in our previous article.^[9]

Preparation of the Colloidal Silica-Fixed and Propionylated Wood (CSPW) Composites

The CSPW composites were prepared as described in our previous article.^[9] However, the specimens used for measuring strength properties, after being propionylated, were immersed in water at reduced pressure for 1 h and leached in flowing water for 7 days. Then they were oven-dried at 105°C for 48 h. The lower drying temperature was used because 120°C might cause strength loss.

Evaluation of the Wood Composites

Weight Percent Gain

The weight percent gain following propionylation (WPG_p) of the CSPW composites was calculated as described previously.^[9]

Oxygen Index

The oxygen index method tests the flame resistance of the composite specimens was the Japanese Industrial Standard JIS K 7201-1976 using an ONI meter (SUGA TEST INSTRUMENTS ON-1). The specimens were burned in an oxygen-nitrogen gas mixture, where the ratio could be controlled. The minimum volume percent concentration of oxygen that will just support

the flaming combustion of specimens is described as the critical oxygen index (OI).

Modulus of Static Elasticity and Bending Strength

The modulus of static elasticity and the bending strengths of specimens were obtained from static bending tests by means of an autograph (Shimadzu AG-5000A) using a 280 mm span length and a 10 mm/min head speed.

Measurement for the Color Changes

The color of the cross sections of specimens before and after treatment was measured by a spectrophotometer (MINOLTA CM-3700d) according to the CIE $L^*a^*b^*$ color system. Five specimens were used for each treatment and three areas (5×5 mm) were measured for each specimen. The color difference of the specimens was calculated according to Equation (1):

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

where ΔE^* is the color difference, ΔL^* is the lightness difference, and Δa^* and Δb^* are the chroma differences.

Fungal Decay Resistance

Fungal decay tests were conducted according to the Japan Wood Preserving Association (JWPA) Standard No.3-1979. Test fungi were a white-rot fungus *Trametes versicolor* (L.: Fr.) Pilát and a brown-rot fungus *Fomitopsis palustris* (Berk. et Curt.) Gilbn. & Ryv. Test specimens were sterilized in gaseous propylene oxide after measuring the initial dry weight. For each set of conditions 3 specimens were placed on a mycelium mat of test fungus in glass jars. The specimens in the glass jars were incubated at 26°C for two months. The extent of fungal attack was evaluated on the basis of the percentage weight loss of the specimen.

RESULTS AND DISCUSSION

Flame Resistance of CSPW Composites

The oxygen index (OI) test is a good method for evaluating the flame resistance of polymer materials with the advantages that it has good repeatability and uses very small samples.^[10,11]

Figure 1 shows the effect of the weight percent gain from colloidal silica fixation (WPG_{csi}) on the oxygen index (OI) of CSPW composites. The propionylated-only treated wood ($WPG_{\text{csi}} = 0$) showed an OI value

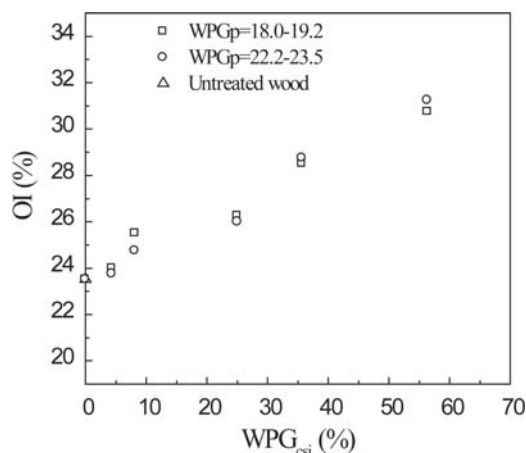


Figure 1. The effect of the weight percent gain from colloidal silica fixation (WPG_{csi}) on the oxygen index (OI) of CSPW composites.

approximately equal to that of the untreated wood, indicating that propionylation does not endow wood with flame resistance as previously reported.^[12,13] However, the CSPW composites showed much higher OI values than the untreated or propionylated wood, and the OI values increased with an increase in WPG_{csi} . This means the CSPW composites are flame resistant due to the colloidal silica treatment.

Mechanical Property of CSPW Composites

In general, long, high-temperature treatments of wood reduce the mechanical strength properties. For this reason, the treatment temperature in these experiments was as low as possible and the treatment period was as short as possible.

Figure 2 shows the modulus of elasticity (MOE) and modulus of rupture (MOR) of the CSPW composites. The results show that all of the treated composites had little reduction in bending strength compared with the untreated wood specimens. The preparation of wood-silicate compound composites made by water glass and other reactant impregnation of wood may lead to a reduction in some of the mechanical properties because of the soaking process in the alkaline solutions of water glass.^[5,6] In this study, the colloidal silica suspension for impregnation was neutralized by adding aqueous acetic acid to bring the pH to 7.0–7.2. Probably for this reason, the colloidal silica fixed-wood (CSW) composites showed little decrease in their mechanical properties. The propionylated woods, similar to acetylated woods, showed little difference in their

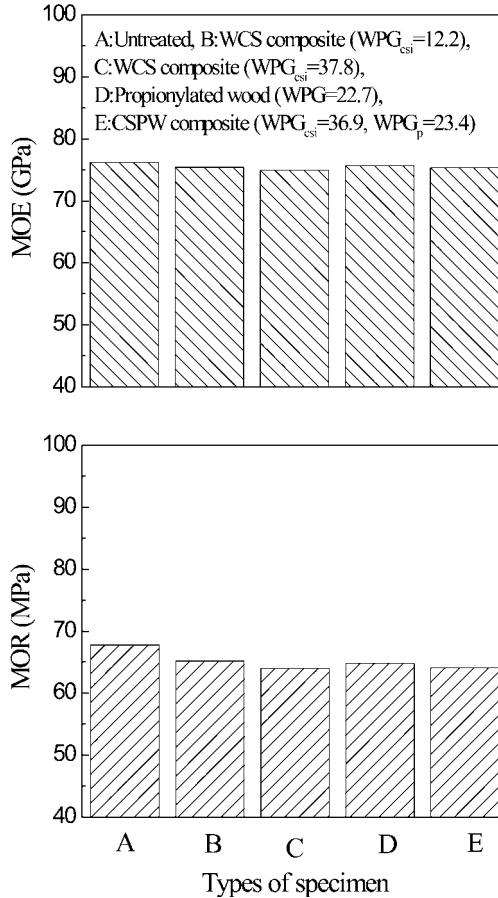


Figure 2. The modulus of elasticity (MOE) and modulus of rupture (MOR) of CSPW composites.

mechanical properties relative to the untreated woods. Therefore, the CSPW composites only exhibited slight reductions in their mechanical strength properties.

Color Change of CSPW Composites

Figure 3 shows the color changes of the CSW and CSPW composites. The composite with a zero WPG_{csi} value means the untreated wood in the CSW composites or the propionylated wood in the CSPW composites.

The CSW composites basically kept the natural color of the wood, probably because of the mild treatment conditions. In fact, the CSW composites got a slight increase in lightness, which may be of advantage to their end-use.

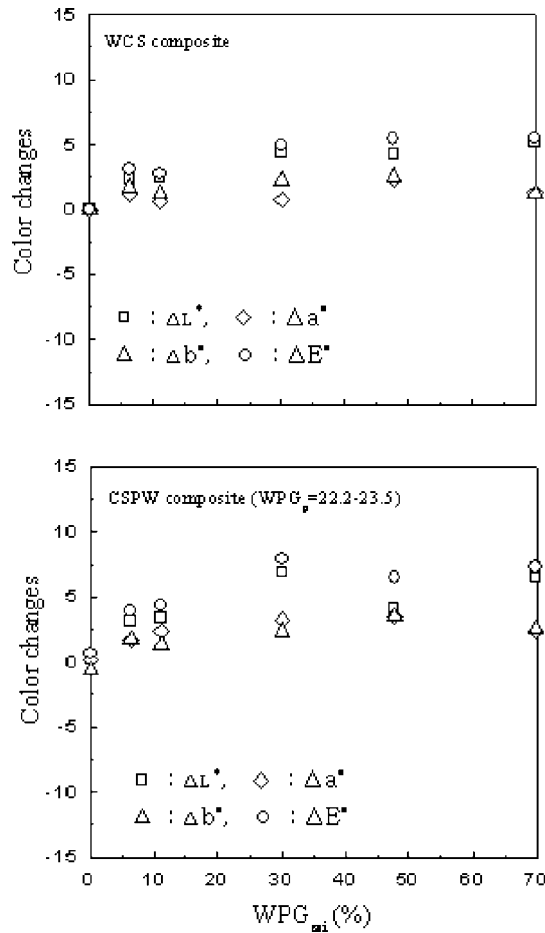


Figure 3. The effect of the weight percent gain from colloidal silica fixation (WPG_{csi}) on the color changes of colloidal silica fixed-wood (CSW) composites and CSPW composites.

The CSPW composites were similar to the CSW composites; little color change occurred following the dual treatment, resulting in wood that essentially retained its natural color.

Fungal Decay Resistance of the Composites

Figure 4 shows the percentage weight losses (WLs) of different wood specimens due to fungal attack by *T. versicolor* or *F. palustris* for 2 months. The results showed that silica gels gave wood some decay resistance,

A: Untreated wood, B: CSW composite ($WPG_{csi}=11.2$), C: CSW composite ($WPG_{csi}=47.8$),
 D: Propionylated wood ($WPG=10.01$), E: Propionylated wood ($WPG=18.3$),
 F: CSPW composite ($WPG_{csi}=47.2, WPG_p=10.1$), G: CSPW composite ($WPG_{csi}=46.9, WPG_p=19.1$)

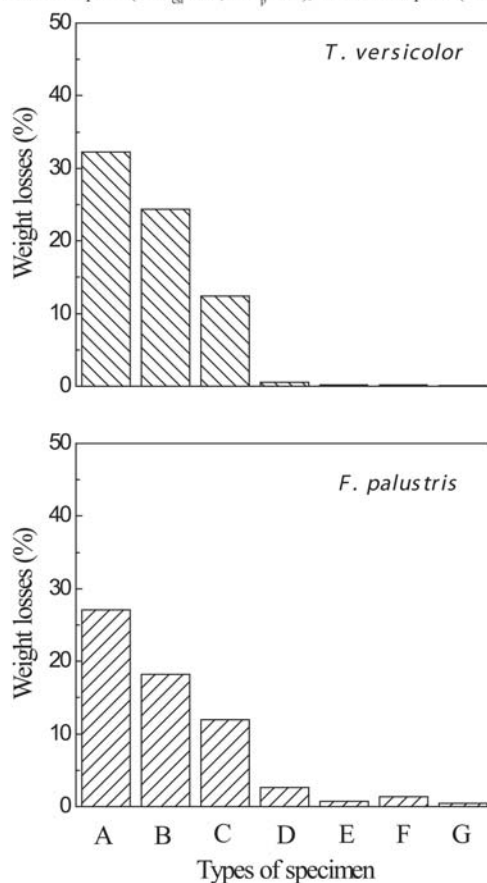


Figure 4. The percentage weight loss of different wood specimens from fungal decay.

and propionylation of the hydroxyl groups provided wood with good decay resistance, as was previously found with acetylation.

CONCLUSIONS

The results of this study lead to the following conclusions: (1) silica fixation can impart flame resistance to wood, (2) wood subjected to silica fixation and propionylation-dual treatment still retains its natural color and mechanical strength properties, and (3) silica fixation and propionylation can provide wood with good decay resistance.

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