

Potential utilization of sodium fluoride (NaF) as a biocide in particleboard production

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Abstract The feasibility of sodium fluoride (NaF) incorporation as a biocide in the manufacture of particleboard was examined. Laboratory-scale particleboards prepared from untreated wood particles were incorporated with NaF powder at target retentions of 1, 1.5 and 3% of the total particle weight. An in-line treatment method was used to introduce the biocide during the blending stage just before adhesive application. Standard static bending and water resistance (water absorption and thickness swelling) tests indicated that embedding of the powder biocide up to the 3% level did not cause any detrimental effects on mechanical and physical properties. The laboratory decay and termite resistance tests showed that even the lower retention levels of 1 and 1.5% NaF were enough to suppress fungal and termite activity and significantly reduce the mass loss and consumption rate values of the specimens when compared to the untreated controls. Spectrophotometric analysis of leachate waters and the mass losses of the leached specimens revealed the tendency of the NaF to be depleted from the composite specimens. Therefore, the tested biocide was found to be appropriate for interior or protected above-ground outdoor exposure conditions.

Keywords Wood-based composites · Biodegradation · In-process treatments · Termite resistance · Sodium fluoride

Introduction

Over the past few decades, wood-based composites have gained broad acceptance for constructional applications, and due to the depletion of the supply of high-quality wood, wood-based composites have been increasingly produced and utilized to replace solid wood. However, since their major component is solid wood, these composites require protection from decay fungi and wood-destroying insects if used in outdoor applications. It has been reported that these composites are experiencing failure due to the abovementioned biological agents [1–4]. Therefore, the durability of wood-based composites remains an important factor for increasing the service life of these composites in challenging environments. Amongst the various pre-treatment or post-treatment techniques, the in-line (or in-process) treatment, which incorporates the biocide during the manufacturing process, has become the method of choice for particleboard and other composites with relatively small particle or fiber size [5, 6]. Compared to the other methods, in-line treatment provides several advantages, including full protection of the composite cross-section and the ability to cut or drill the composite at any time without applying remedial treatments. In recent years, several biocides such as zinc borate, boric acid and permethrin have been utilized for the in-process treatment of particleboards and medium-density fiberboards (MDFs) [7–10]. Sodium fluoride (NaF) has been used effectively for many years in Europe and the USA as a registered wood preservative chemical for solid wood applications (e.g., railroad ties and utility poles) against wood decaying fungi and subterranean termites. In Australia and USA fluoride rods (e.g., fluroids, 98% sodium fluoride, Osmose Utilities Services, Inc.) were used as internal remedial treatments in utility poles [11, 12]. It is not very soluble in

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water but has poor resistance to leaching. The first multi-salt preservative chemical was patented by Dr. Wolman in Germany and consisted of sodium fluoride (85%), dinitrophenol (10%) and sodium dichromate (5%) known as Triolith [13, 14]. Because their high efficacy against decay fungi, fluorides became an important wood preservatives in initial and remedial treatments due to their ability to diffuse via free water through refractory wood species [15]. Bifluorides were also used as wood preservatives in Germany against decay fungi and some insect species especially European House Borer, *Hylotrupes bajulus* (L.). As the previous literature mostly deals with biological performance of NaF or other fluoride salt treated solid wood, the current study focuses on NaF treatment of wood-based composites via in-line treatment. Additionally, NaF is an incombustible, colorless, odorless, non-corrosive crystalline solid or powder with very high melting (993 °C) and boiling (1704 °C) temperatures, which makes it suitable for high-temperature manufacturing processes such as hot pressing or extrusion. As a medication, it is primarily used to prevent tooth decay and osteoporosis in humans [16]. Thus, as a biocide, it might be considered as having a minimal environmental impact with low mammalian toxicity when compared to others. However, the efficacy of NaF against wood decay fungi and termites in wood-based composite production is poorly documented and requires further investigation. The objectives of this paper were to evaluate the feasibility of sodium fluoride (NaF) as a biocide for in-line treatment during the manufacture of particleboard and to investigate its efficacy, leachability and effects on physical and mechanical properties.

Materials and methods

Board manufacturing

Wood particles obtained in Japan from demolished construction materials were used for the board manufacturing. The particles consisted of untreated, mixed wood species having a moisture content of 6–7%. Finely powdered NaF (99% purity, from Nacalai Tesque Inc., Kyoto, Japan) was introduced into the blender. Four different target retentions (0, 1, 1.5 and 3%) of the total particle weight were chosen for this study. The wood particles and finely powdered NaF were mixed for 60 s and then a polymeric diphenylmethane diisocyanate (pMDI) resin at the level of 10% of the total wood-particle weight was sprayed onto the mixture. A 300 × 300 mm forming box was used to prepare the board mats before hot pressing. Single-layer mats were hot pressed at 160 °C for 10 min. The target thickness and density of the boards were 15 mm and 700 kg/m³, respectively. No other chemical was used during

manufacturing and three replicates were conducted for each retention level, totaling 12 boards including 0% NaF controls. The specimens for the bending, decay and termite tests were cut from those boards after 3 weeks of conditioning at 22 °C and 60% relative humidity (RH).

Leaching test

A robust leaching procedure was applied to additional sets of specimens to test the leachability of NaF and to assess the decay and termite resistance after leaching. The specimens were exposed to 10 cycles of leaching by immersion in deionized water at 10 times the amount of the specimen volume. The water was stirred with a magnetic stirrer at 400–450 rpm for 8 h. The specimens then underwent a 16-h drying period in a circulating-type oven at 60 °C. A large leaching-water volume was intentionally used to create an aggressive leaching environment. During the 10-day leaching period, 20 ml of leachate was taken from each leaching flask and replaced with an equal amount of fresh deionized water at time intervals of 24, 48, 72, 120, 168 and 240 h as per JIS K 1571 [17]. The leachate samples were then stored in a refrigerator at 4 °C until the spectrophotometric analysis for fluorine content.

Mechanical tests

The Japanese Industrial Standard [18] for particleboards was followed to determine the static bending properties, modulus of rupture (MOR) and modulus of elasticity (MOE) of the manufactured particleboards. Six 230 (L) × 30 (W) × 15 (T) mm bending specimens for each retention group (two replicates for each board) were tested over a span of 180 mm and a crosshead speed of 10 mm/min with an Instron universal testing machine (Instron Model 4411, Norwood, MA, USA). An analysis of variance [19] test was used to compare the effects of the NaF retention levels on the bending properties of the manufactured particleboards.

Water absorption and thickness swelling

The water absorption (WA) and thickness swelling (TS) tests were also conducted according to JIS A 5908 [18] with minor modifications for specimen size. For each retention level, five replicates were conducted on the 30 × 30 × 15 mm specimens. The thickness at the center of each specimen was recorded along with its weight before immersing the test pieces 3 cm below the water surface at 22 °C, and then at 2- and 24-h intervals after immersion to determine the short- and long-term water resistance properties, respectively.

Determination of NaF in leachate water

A leachate sample (20 ml) filtered through a 0.45 µm cellulose membrane was mixed with perchloric acid and distilled at 140–150 °C. The fluorine content in the distillate was determined by the lanthanum–alizarin complexone method described in Japan Industrial Standard K 0102 34.1 [20] using a UV–VIS spectrophotometer (V-530, JASCO, Tokyo, Japan) at the Toray Research Center Inc., Tokyo, Japan.

Laboratory decay and termite resistance tests

Eighteen specimens (nine 30 × 30 × 15 mm unleached and leached) were prepared for the decay tests. The decay tests were performed according to the Japanese Industrial Standard JIS K 1571 [17]. Sugi (*Cryptomeria japonica* (L.f.) D. Don) sapwood specimens were also prepared according to JIS size as the reference material. All specimens were oven-dried at 60 ± 2 °C for 48 h before their pre-decay weights were recorded, and sterilization was performed under vacuum with gaseous ethylene oxide for 18 h. Three specimens were kept in glass jars for 12 weeks in the dark at 26 ± 2 °C exposed to a monoculture of either the white rot fungus *Trametes versicolor* (L:Fr.) Pilat [Forestry and Forest Products Research Institute of Japan (FFPRI) 1030] or the brown rot fungus *Fomitopsis palustris* (Berk et Curt.) Gilb & Ryv (FFPRI 0507). The nutritional media consisted of glucose (2%), peptone (0.15%), malt extract (0.75%) and distilled water (97.1%) and was absorbed into white quartz sand. For the brown rot test, all specimens were raised 1 mm above the sand surface using plastic meshes. Three decay jars were used to test nine replicates for each particleboard group against each decay fungus. The total number of composite specimens was 144, representing the four different retention levels (including the 0% NaF controls) and two leaching conditions (leached and unleached). In addition, 18 sugi reference specimens were tested. At the end of the decay tests, immediately after removal of the surface mycelium, the specimens were weighed and the post-decay moisture contents were calculated. A further weight measurement took place after the specimens were oven-dried at 60 ± 2 °C for 48 h to determine their mass loss.

Three specimens were randomly selected from each treatment group for the laboratory termite tests. The total number of termite test specimens was 15 including the sugi sapwood reference material. Each specimen was placed at the center of the plaster bottom of a cylindrical test container 6 cm in depth × 8 cm in diameter. A total of 150 workers and 15 soldiers of *Coptotermes formosanus* Shiraki were collected from a laboratory colony, which was harvested from Okayama Prefecture and had been

maintained for more than 10 years in the Deterioration Organisms Laboratory (DOL) of RISH at Uji campus of Kyoto University. The collected termites were immediately transferred into each test container according to JIS K 1571 [17]. The assembled containers were placed on a water-moistened cotton pad and stored in the dark at 28 ± 2 °C and a RH of more than 80% for 3 weeks. The percent of mass loss (mass difference at the end of the test), termite mortality (number of live termites at the end of the test) and consumption rates (the amount of the composite consumed per worker-termite day during the test period) were also determined.

Results and discussion

Based on the spectrophotometric analysis of leachate water samples during the leaching test, the *F* was depleted over a period of 240 h. The leaching amount depended on the retention levels and the leaching time (Fig. 1). At the end of the leaching period, the *F* concentration in the leachate water had been reduced to below 50 ppm for all retention levels. Similarly, Pan et al. [12] reported that only 34.05% of the fluorine remained in 2% NaF-treated wood blocks exposed for 4 weeks in an underground leaching test in Hangzhou, China. However, almost all the fluorine was lost from NaF-treated wood blocks at the end of a 312-h water-contact leaching process based on the AWWPA E 10 standard [21]. This indicated that NaF leachability is dependent on the leaching procedure and the standard used. In addition, NaF has a relatively high solubility when compared other biocides utilized in wood-based protection such as zinc borate. Tascioglu et al. [10] reported that in 2% zinc borate (ZnB) incorporated particleboards exposed to the same leaching process, the B content was reduced by 34.4%, while the Zn content remained unchanged.

Table 1 shows the physical properties of oven-dried density and moisture content at the time of testing and the mechanical properties of MOR and MOE of the boards processed for this experiment. The oven-dried density of the boards (0.62–0.71 g/cm³) was within the targeted density range of the final product. According to the experimental data, incorporation of NaF up to the 3% level did not negatively affect the MOR and MOE values of the manufactured boards. Based on statistical analysis, no significant differences were found between the mechanical test values of the untreated controls and those of the biocide-incorporated boards nor among the three different retention groups, with *p* values of 0.738 and 0.721 for MOR and MOE, respectively. The moisture content (MC) analysis also indicated that all specimens were equilibrated in the same moisture group before testing (*p* value = 0.248) which eliminated any moisture content

Fig. 1 Changes of fluoride ion concentration in leachate waters from 3 different retentions over 240 h of leaching

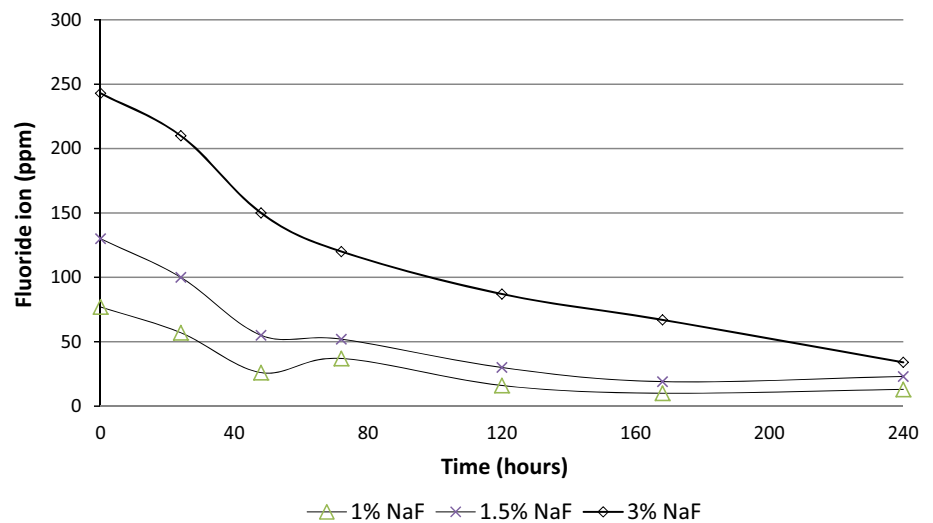


Table 1 Physical and mechanical properties of sodium fluoride (NaF) incorporated particleboards (mean of 6 specimens—values in parentheses are standard deviations)

| NaF (%) | Oven-dried density (g/cm ³) | MC ^a (%) | MOR ^b (MPa) | MOE ^c (MPa) |
|-----------------|---|---------------------|------------------------|------------------------|
| 0.00 | 0.62 | 6.15 (0.31) a | 22.55 (6.34) a | 2499 (522) a |
| 1.00 | 0.71 | 6.05 (0.19) a | 20.50 (3.64) a | 2318 (301) a |
| 1.50 | 0.65 | 6.30 (0.13) a | 21.40 (3.85) a | 2419 (355) a |
| 3.00 | 0.67 | 6.12 (0.16) a | 23.05 (2.58) a | 2551 (253) a |
| <i>p</i> values | | 0.248 | 0.738 | 0.721 |

Mean within each column followed by different letters are significantly different (*p* ≤ 0.05)

^a MC (moisture content) at the time of mechanical testing

^b Modulus of rupture

^c Modulus of elasticity

Table 2 Water resistance properties of sodium fluoride (NaF) incorporated particleboards (mean of 5 specimens—values in parentheses are standard deviations)

| NaF (%) | Water absorption (%) | | Thickness swelling (%) | |
|-----------------|----------------------|-----------------|------------------------|----------------|
| | 2 h | 24 h | 2 h | 24 h |
| 0.00 | 10.02 (2.01) a | 27.78 (5.60) a | 4.26 (0.47) a | 9.28 (0.94) a |
| 1.00 | 15.92 (3.32) a | 49.64 (8.84) b | 5.84 (0.97) a | 12.46 (1.15) b |
| 1.50 | 14.48 (2.70) a | 41.14 (8.83) b | 5.44 (1.01) a | 12.44 (2.23) b |
| 3.00 | 15.94 (5.82) a | 41.38 (11.94) b | 5.28 (1.02) a | 11.68 (2.29) b |
| <i>p</i> values | 0.075 | 0.013 | 0.076 | 0.036 |

Mean within each column followed by different letters are significantly different (*p* ≤ 0.05)

effect on the mechanical properties. Vick et al. [22] reported that NaF treatment did not interfere with the adhesion of phenol–formaldehyde adhesive on poplar veneers at retentions of 3.2–9.6 kg/m³. Although no specific report has been found dealing with the effects of NaF addition on the mechanical properties of wood-based composites, these findings were in accord with previous studies in the literature indicating that retention levels of other biocides of up to 1.5% (boric acid) [7] and 2% (zinc borate) [4, 23] had no adverse effects on mechanical properties.

WA and TS values, as seen in Table 2, indicated that there were no statistically significant differences between the untreated controls and the three different retention levels at the end of the 2-h testing period, with *p* values of 0.075 and 0.076, respectively. On the other hand, when the time was advanced to 24 h, significant differences were detected for both WA and TS tests. The WA percent values increased from approximately 27% to a mean of 44%, depending on the NaF content. The TS values also increased from 9 to 12% (for 1% NaF), 12% (for 1.5% NaF) and 11% (for 3% NaF). The rising trend was not

Table 3 Mean percent mass loss of sodium fluoride (NaF) incorporated particleboards exposed to *Trametes versicolor* and *Fomitopsis palustris* for 12 weeks (mean of 9 specimens—values in parentheses are standard deviations)

| NaF (%) | <i>T. versicolor</i> | | <i>F. palustris</i> | |
|-------------------|----------------------|----------------|---------------------|----------------|
| | Unleached | Leached | Unleached | Leached |
| 0.00 | 14.6 (2.81) b | 15.3 (7.02) c | 20.8 (3.36) b | 17.3 (4.44) a |
| 1.00 | 2.8 (0.21) a | 11.2 (3.89) b | 1.8 (0.20) a | 17.6 (10.40) a |
| 1.50 | 3.0 (0.18) a | 12.0 (2.73) bc | 2.3 (0.15) a | 16.4 (8.78) a |
| 3.00 | 3.9 (0.15) a | 5.1 (3.43) a | 3.0 (0.23) a | 5.7 (11.60) b |
| Sugi ^a | 31.8 (3.70) | – | 41.4 (3.54) | – |
| <i>p</i> values | 0.000 | 0.000 | 0.000 | 0.065 |

Means within each column followed by different letters are significantly different ($p \leq 0.05$)

^a Sugi sapwood was used as untreated reference material and excluded from statistical analysis

Table 4 Mean percent mass loss [ML (%)], mortality [M (%)] and consumption rate [CR ($\mu\text{g}/\text{termite}/\text{day}$)] of NaF-incorporated particleboards exposed to *Coptotermes formosanus* for 3 weeks (mean of 3 specimens—values in parentheses are standard deviations)

| NaF (%) | Unleached | | | Leached | | |
|-------------------|--------------|----------------|----------------|--------------|---------------|-----------------|
| | ML | M | CR | ML | M | CR |
| 0.00 | 8.2 (0.68) b | 24.2 (3.32) a | 88.5 (12.81) b | 8.3 (0.98) a | 30.0 (4.39) a | 96.7 (2.36) b |
| 1.00 | 2.3 (0.05) a | 99.3 (1.15) b | 48.2 (8.88) a | 6.4 (1.86) a | 27.5 (7.60) a | 75.8 (14.00) a |
| 1.50 | 2.1 (0.32) a | 99.8 (0.40) b | 42.5 (4.64) a | 7.1 (1.03) a | 40.9 (1.01) b | 85.3 (10.67) ab |
| 3.00 | 2.2 (0.05) a | 100.0 (0.00) b | 48.2 (6.04) a | 5.4 (0.32) a | 42.4 (5.36) b | 71.2 (2.89) a |
| Sugi ^a | 17.3 (2.98) | 39.1 (4.02) | 100.4 (9.68) | – | – | – |
| <i>p</i> values | 0.000 | 0.000 | 0.001 | 0.086 | 0.016 | 0.035 |

Mean within each column followed by different letters are significantly different ($p \leq 0.05$)

^a Sugi sapwood was used as untreated reference material and excluded from statistical analysis

linearly in order with the biocide retention levels for either test. The incorporation of NaF, which is known to be a hygroscopic salt [16], might have contributed to the increased WA and TS values. According to the Engineered Wood Products Association of Australasia [24], the 24-h TS value of 12% for the treated composites fell within the acceptable range for standard particleboards with thicknesses of 13–22 mm and a density range of 0.66–0.68 g/cm³.

As shown in Table 3, the untreated sugi sapwood reference material was severely consumed by both test fungi, revealing the high fungal activity under the test conditions. Both unleached and leached control specimens resulted in an approximate mass loss of 15% under *T. versicolor* exposure. Similarly, *F. palustris* caused approximate mass losses of 21 and 17%, respectively, for the unleached and leached control specimens at the end of the same exposure time. When the unleached specimens were considered, the mass losses were significantly suppressed from around 15 to 2–4% for *T. versicolor*, and from around 21 to 2–3% for *F. palustris* with the addition of NaF up to 3% by total particle weight. This phenomenon indicated that the NaF was effective against both test fungi under non-leach environments. However, the efficacy of NaF was adversely affected by the robust leaching procedure. In particular, the lower retentions (1 and 1.5%) gave mass loss values similar to those of the untreated controls for both fungal species. Only the 3% retention level maintained some level of anti-

fungal effect, with mean mass losses of 5.1 and 5.7% for *T. versicolor* and *F. palustris*, respectively. Previous research in the literature has noted that a retention of 0.33 kg/m³ F (as fluoride) was required as a threshold value against the two brown rot fungi tested [25]. The relatively high solubility of sodium fluoride (4.0 g/100 ml water solubility at 20 °C) and the robust leaching procedure might be considered as the major contributors to the higher mass losses of the leached specimens. When both fungi were considered, statistical analysis did not reveal any linear relationship regarding mass loss values amongst the three different retention levels.

In general, the laboratory termite resistance of the NaF-incorporated particleboards was greater than that of the untreated control samples under unleached conditions. As the NaF content increased, the mass losses of the treated boards were significantly reduced. The 3% NaF addition, for example, showed a reduction in mass loss of approximately 73% and a reduction in consumption rate by *C. formosanus* of approximately 46%. Even the lowest retention level (1%) was able to suppress mass loss by less than 3% against the laboratory termite activity. Similarly, the mortality rates significantly increased from approximately 24 to 100%. The efficacy of the biocide seemed to be depleted by the leaching process since the mass loss values did not show the same trend as the unleached series. Although some reductions in mass loss were recorded (8.3–5.4% for the highest retention), they were not found to

be statistically significant (p value = 0.086) due to the high variation in the standard deviations. On the contrary, based on ANOVA analysis, significant reductions were found in the mortality and consumption rates for the leached specimens when the NaF retention was increased (Table 4).

Conclusion

The incorporation of NaF via in-process treatment was demonstrated to be feasible for the protection of particleboards from biological agents in unleached environments, with no adverse effects on mechanical or physical properties. When used in indoor or protected above-ground (unleached) conditions, even the addition of NaF at the lowest retention level (1%) resulted in significant reductions in the mass loss values caused by the decay fungi and termite species tested. However, the relatively high leachability of NaF in water should be taken into account if outdoor or water/ground contact applications are involved. The lower NaF retention levels after the leaching process implied that the remaining biocide was insufficient in suppressing decay and termite activity in the treated particleboards, and thus, higher retentions might be required to achieve lower mass loss and consumption rates.

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