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Won-Joung Hwang · S. Nami Kartal · Yuji Imamura
Kunio Tsunoda · Katsumi Shinoda

Comparative effectiveness of two alkylammonium compounds as wood preservatives

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Abstract This study describes a laboratory evaluation of the efficacy of two alkylammonium compounds [didecyldimethylammonium tetrafluoroborate (DBF) and didecyldimethylammonium chloride (DDAC)] when applied via vacuum impregnation or superficial treatment. Treated wood specimens were tested for their termite and microbial resistance under controlled laboratory conditions. The higher chemical retentions were needed to suppress the feeding by *Coptotermes formosanus* ≦3% mass loss in the multichoice test than in the no-choice test. The DBF and DDAC retention levels necessary to meet the performance requirement ≦3% mass loss after 12-week fungal exposure varied with wood species. The retention level of 3 kg/m^3 for DBF and DDAC was generally high to keep the nondurable wood species free of decay. Although there was no difference between DBF and DDAC in the efficacy against decay and termite attack, the former slightly outperformed the latter as an antimold and antisapstain agent.

Key words Alkylammonium compound · Subterranean termite · Decay fungus · Mold · Staining fungus

Introduction

Although there are variations in natural durability between and within the heartwoods of individual trees and the heartwood of some wood species can resist wood-degrading organisms,[1] most of the commonly used wood species are moderately durable or nondurable. Wood put to commercial service often contains both sapwood and heartwood portions within a given structure. As a result, the retention of preservative compounds used to prevent biodegradation, the fixation of chemicals, and the leaching of active ingredients, as well as the resistance of treated sapwood and heartwood against wood-degrading organisms are thought to differ.[2] Because it is commonly known that the sapwood portion is more susceptible to biological attacks than the heartwood portion of the same wood species, the application of preservative treatment has been well accepted for the sapwood.

Boron has been an important element in some widely used wood preservatives. Despite many advantages of boron wood preservatives, boron itself does not remain fixed in wood and as a result it cannot protect wood that experiences ground contact and or unprotected outdoor conditions. On the other hand, some research has been performed in an attempt to improve the resistance of boron-containing wood preservatives to leaching and other disadvantageous properties of treated wood.[3]

A novel alkylammonium compound (AAC), didecyldimethylammonium tetrafluoroborate (DBF), which contains boric tetrafluoride (BF_4^-), as a counter ion, has been recently developed. Our previous studies demonstrated that DBF was effective against the subterranean termite *Coptotermes formosanus* at a retention level of 4 kg/m^3 even after 10 cycles of leaching and evaporation.[4] We also found in a laboratory termite test that DBF treatments needed higher retention levels in nondurable wood species than in durable wood species in order to ensure protection.[4–7] In this study, the preservative efficacy of DBF was compared with that of didecyldimethylammonium chloride (DDAC) at several retention levels in wood species of differing natural durabilities. Efficacy was evaluated in terms of the choice termite feeding test, a decay test, and a mold and sapstain fungus test.

W.-J. Hwang (✉) · Y. Imamura · K. Tsunoda
Research Institute for Sustainable Humanosphere (RISH), Kyoto University, Gokasho, Uji 611-0011, Japan
Tel. +81-774-38-3663; Fax +81-774-38-3664
e-mail: wjhwang@rish.kyoto-u.ac.jp

S.N. Kartal
Forestry Faculty, Istanbul University, Bahcekoy 34473, Istanbul, Turkey

K. Shinoda
Sanyo Chemical Industries, Ltd., Kyoto 605-0995, Japan

Materials and methods

Preparation of wood specimens

Two sizes of wood specimens were used for laboratory biological tests. Wood specimens measuring 20 (R) × 20 (T) × 10 mm (L) were prepared from the heartwood of *Chamaecyparis obtusa* Endl., *Cryptomeria japonica* D. Don., *Pseudotsuga menziesii* (Mirbel) Franco, *Tsuga heterophylla* Sarg., and the sapwood of *C. japonica* and *Fagus crenata* Blume for both termite and decay tests. The oven-dry density of each wood specimen was determined after drying at 105° ± 2°C for 24 h. Wood specimens, 3 (R) × 20 (T) × 50 mm (L), were prepared from the sapwood of *C. japonica* and *F. crenata* for the mold and sapstain fungi tests.

Treatment of wood specimens

Wood specimens were treated in the same manner previously applied to the no-choice termite test.[4] These specimens were pressure (vacuum)-impregnated with aqueous solutions of either DDAC or DBF at 0.01, 0.10, 0.50, and 1.0% (m/m) in order to obtain the respective target retentions of 0.06–0.08, 0.60–0.80, 3.00–4.00, and 6.00–8.00 kg/m^3 varying with wood species for the decay test. Because treatment concentrations were common to different wood species, the calculated retentions varied among wood species at the same treatment concentration. The treated wood specimens were reweighed to determine retentions of DBF or DDAC after using a paper towel to remove excessive treatment solution from the wood surfaces. All treated specimens were then stored at ambient conditions for at least 20 days until they were exposed to weathering cycles. A selected retention level for each wood species was evaluated in the choice termite feeding test.

Superficial (brush-on) treatments with DBF or DDAC at a rate of 160 g/m^2 (treatment solution/wood surface)[8] were conducted to evaluate the resistance of the treated wood against mold and sapstain fungi.

Weathering process

The treated wood specimens used for the termite and decay tests were weathered according to JIS K 1571-2004.[9] This process involved immersing the wood specimens in distilled water (ten volumes of distilled water to one volume of wood), stirring with a magnetic stirrer (400–450 rpm) at 26° ± 2°C for 8 h, followed by drying at 60° ± 2°C for 16 h. This cycle was then repeated nine times. Water was renewed at every weathering cycle.

Multichoice termite feeding test

The DBF retentions were 0.72, 0.68, 3.44, 3.25, 4.03, and 6.18 kg/m^3 for the heartwood of *C. obtusa*, *C. japonica*, *T. heterophylla*, and *P. menziesii*, and the sapwood of *C. japonica* and *F. crenata*, respectively. The selected retentions for DDAC were 0.07, 0.60, 3.06, 6.04, 8.09, and 3.12 kg/m^3 for the wood species in the same order as DBF mentioned above. Untreated wood specimens prepared from the heartwood of *C. obtusa* and *C. japonica* were also included in the test as reference materials to compare the efficacy of DBF and DDAC treatments. The oven-dried weights of all specimens were measured after 3 days of drying at 60° ± 2°C. The multichoice feeding test was conducted as follows. Eight wood specimens consisting of one each of the six treated wood species, and untreated *C. obtusa* and *C. japonica* heartwood were placed in a plastic container (150 mm in diameter) with a lid together with 50 g of vermiculite and 90 ml of distilled water. A total of 1000 workers and 100 soldiers of *Coptotermes formosanus* Shiraki, collected from a laboratory colony maintained at the Research Institute for Sustainable Humanosphere of Kyoto University, were introduced into each test container. Three replicates were assayed against termites. The assembled containers were kept at 28° ± 2°C and 70%–80% relative humidity (RH) in a darkroom for 4 weeks. The recovered wood specimens were carefully cleaned of debris using tap water and oven-dried at 60° ± 2°C for 3 days. The percent of mass loss of each wood specimen caused by termite attack was calculated based on the difference in oven-dried weights before and after the multichoice feeding test.

Three separate test assemblies were prepared using only the untreated wood specimens of the above-mentioned wood species. Each test assembly contained one of each of the wood species and was handled in the same manner as the eight wood specimens. To compare the results, all data were analyzed by one-way analysis of variance (ANOVA) (Excel 2000, Microsoft), and mass losses of each wood species were then statistically compared by Tukey's test.[10]

Decay test

The decay tests were conducted according to JIS K 1571-2004[9] using a monoculture of either the brown-rot fungus, *Fomitopsis palustris* (Berk. et Curt.) Gilbn. & Ryv. (FFPRI 0507), or the white-rot fungus, *Trametes versicolor* (L.: Fr.) Pilat (FFPRI 1030), with a minor modification in the proportion of nutrient constituents (a half concentration of each component for *F. palustris*).

The dry weights of both weathered and unweathered wood specimens were measured first after drying at 60° ± 2°C for 3 days and then sterilized with gaseous ethylene oxide. Three wood specimens having undergone the same treatment were placed on the surface of fully grown mycelium of the fungus in a glass jar with a plastic mesh in between the mycelial mat and the wood specimens for *F. palustris*, while plastic mesh was not used for *T. versicolor*. The glass jars were then incubated at 26° ± 2°C and 70%–80% RH for 12 weeks.

Nine replicates of each combination of decay fungus and treatment were tested. The extent of the fungal attack of each treatment group was expressed as mean percent of mass loss based on the difference in the oven-dried weights

of nine individual specimens before and after the decay procedure.

Mold and sapstain fungi resistance test

The microbial test involving mold and sapstaining fungi was principally carried out according to the Japan Wood Preserving Association Standard-2.[11] The only exception was the treatment method of the wood specimens. A brush-on treatment was used in the current experiment instead of the dip treatment prescribed by the standard in order to precisely compare the effect of the two test compounds. Six replicates were prepared for each test fungus and treatment.

The five fungi used were *Aspergillus niger* van Tieghem IFO 6341, *Penicillium funiculosum* Thom IFO 6345, *Aureobasidium pullulans* (de Bary) Arnaud IFO 6353, *Gliocladium virens* Miller, Giddens & Foster IFO 6355, and *Rhizopus stolonifer* (Ehrenberg: Fries) Vuillemin SN 32 IFO 31005.

Fungal inoculation was conducted with spore suspensions of each test fungus. Monocultures of the test fungi were prepared on 2% agar medium in 100-ml Erlenmeyer flasks [2% malt extract, 2.5% glucose, 0.5% peptone, 0.3% potassium phosphate (monobasic), and 0.2% magnesium sulfate]. Fully grown mycelia were collected with a glass rod and water containing 0.005% sodium dioctyl sulfosuccinate was used to prepare the spore suspension.

Three wood specimens of the same treatment group were sterilized with gaseous ethylene oxide, dipped in sterilized distilled water for 5 s, and placed in a polystyrene Petri dish prior to the inoculation with the previously prepared spore suspension of a test fungus. Six replicate wood specimens were used for each combination of treatment, test fungus, and wood species.

The Petri dishes were kept at 26° ± 2°C and 70%–80% RH in darkness for 4 weeks. Microbial growth on the side and upper surface of the wood specimens was rated each week under a binocular microscope. Microbial growth ratings (0–3) are shown in Table 1. To facilitate comparisons of preventive effectiveness among the treatments, the mean rating scores for the five test fungi were totaled to calculate the degree of damage (D) caused by fungal attack according to the equation shown below; where C and T are the mean rating scores of untreated and treated wood specimens for each test fungus, respectively.

$$D = [(\Sigma T_n)/(\Sigma C_n)] \times 100 \quad (n = 1\text{–}5)$$

Results and discussion

Multichoice termite feeding test

The mean amounts of wood eaten were 0.08, 0.30, 0.49, 0.34, 0.55, and 0.77 g for *Cryptomeria japonica* heartwood, *Chamaecyparis obtusa* heartwood, *Fagus crenata* sapwood, *C. japonica* sapwood, *Pseudotsuga menziesii* heartwood, and *Tsuga heterophylla* heartwood, respectively. A comparison of the data obtained by the present multichoice and the previous no-choice termite feeding tests[4] indicated that the multichoice test always resulted in the higher percent mass loss (1.5 to 6.7 times higher) regardless of wood species. This difference in percent mass loss in the two tests remained unexplained because the number of termites per wood specimen and test duration could not be matched for comparison.

Figure 1 shows the mean mass losses of treated and untreated wood specimens after the multichoice feeding test. In similar results to those of the multichoice feeding test involving untreated wood specimens, the untreated heartwood specimens of *C. obtusa* (percent mass loss: 3.35%–7.88%) and *C. japonica* (percent mass loss: 5.79%–8.39%) were eaten more than those in the no-choice feeding test (2.33%, 3.24% mass losses for *C. obtusa* and *C. japonica*, respectively).[4] However, when the wood consumption rate of total wood specimens was calculated based on the assumption that mortality increased linearly toward the end of the test duration, the figure in the multichoice feeding test was 14–22 μg per termite per day, which was not significantly different from the 13–28 μg per termite per day in the no-choice feeding test involving the heartwood of *C. obtusa* and *C. japonica*.[4] This comparison provided strong indication of the termite feeding preference: they selectively took the susceptible materials.[4]

A few treated wood specimens sustained a mass loss of greater than 2%, although in the no-choice test the tested retentions were seen to be high enough to suppress termite attack. These unsuccessful cases included DBF-treated *C. obtusa* heartwood with a retention of 0.72 kg/m^3 and *P. menziesii* heartwood (3.25 kg/m^3), and DDAC-treated *C. obtusa* heartwood (0.07 kg/m^3), *T. heterophylla* heartwood (3.06 kg/m^3), and *F. crenata* sapwood (3.12 kg/m^3). Because *Coptotermes formosanus* termites were more tolerant to copper naphthenate-treated wood in the no-choice test than in the two-choice test,[12] the current results were thoroughly contrary to our expectations. However, the protective efficacy of the tested chemical(s) in regard to their repellence and antifeeding effects might be more important in the deter-

Table 1. Descriptions of rating scores for inhibiting the growth of mold and sapstain fungi on wood specimens

Rating score	Description
0	No fungal growth on the wood specimen
1	Slight attack: fungal growth restricted to only the side face of wood specimen
2	Moderate attack: fungal growth covered less than 1/3 of the upper surface of the wood specimen
3	Heavy attack: fungal growth covered 1/3 or more of the upper surface of the wood specimen

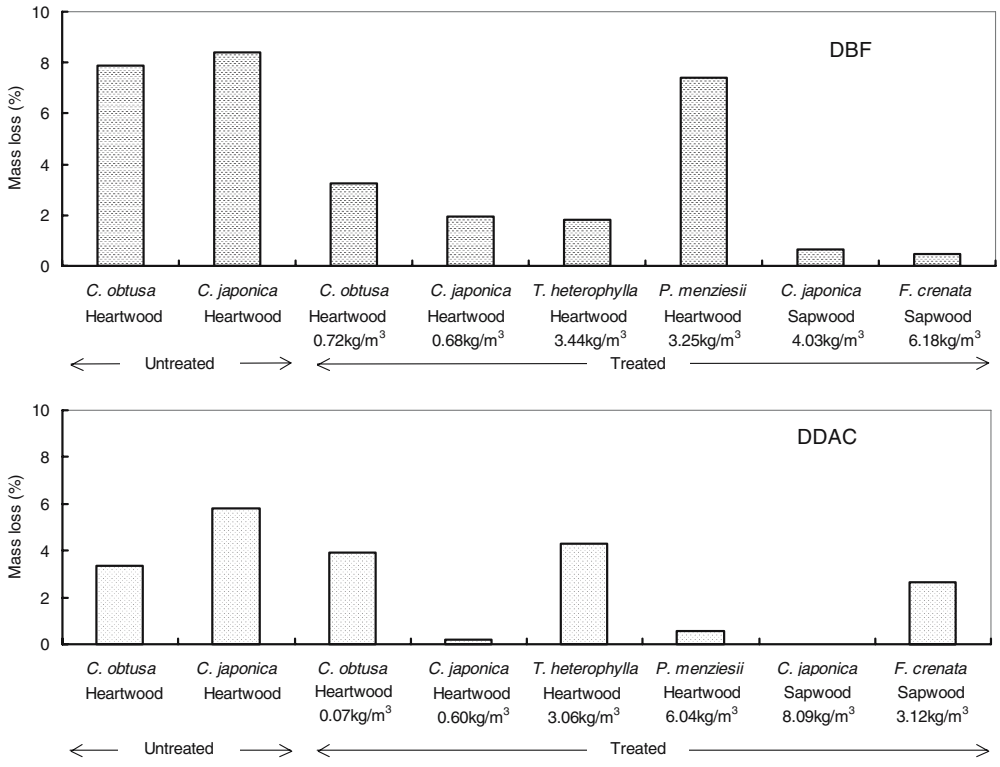

Fig. 1. Mass losses of wood specimens treated (and untreated) with didecyldimethylammonium tetrafluoroborate (*DBF*) and didecyldimethylammonium chloride (*DDAC*) determined by the 4-week choice feeding test with the subterranean termite, *Coptotermes formosanus*

Table 2. Toxic thresholds of alkylammonium compounds (AACs) determined with weathered wood specimens in the laboratory test

Wood species		AAC	Toxic threshold (kg/m^3)			
			F. palustris	*T. versicolor*	*C. formosanus*[a]	Overall
C. japonica	Heartwood	DBF	0.63–3.52	0.63–3.52	<0.06	0.63–3.52
		DDAC	0.06–0.66	0.66–3.38	<0.07	0.66–3.38
C. obtusa	Heartwood	DBF	0.71–3.63	0.71–3.63	<0.07	0.71–3.63
		DDAC	0.67–3.38	0.07–0.67	<0.07	0.67–3.38
P. menziesii	Heartwood	DBF	0.60–3.09	3.09–6.19	0.06–0.60	3.09–6.19
		DDAC	0.60–3.01	3.01–6.09	0.59–2.97	3.01–6.09
T. heterophylla	Heartwood	DBF	0.67–3.30	0.67–3.30	0.06–0.63	0.67–3.30
		DDAC	0.64–3.15	0.64–3.15	0.59–2.97	0.64–3.15
C. japonica	Sapwood	DBF	0.80–4.00	0.80–4.00	0.80–3.99	0.80–4.00
		DDAC	0.80–4.00	0.80–4.00	0.79–3.99	0.80–4.00
F. crenata	Sapwood	DBF	3.20–6.50	>6.50	0.61–3.03	>6.50
		DDAC	3.19–6.34	3.19–6.34	0.61–3.18	3.19–6.34

[a] Figures are based on the previous experimental data[4]

rence of termite feeding,[12] and the results should be interpreted with caution.

It can be concluded from the results of the no-choice and multichoice termite feeding tests that termites might attack wood treated with the tested AACs at the approximate toxic thresholds of some nondurable and moderately durable wood species in the field. Therefore, it would be necessary to practically treat wood at retentions slightly higher than the toxic threshold values determined in the laboratory no-choice or multichoice tests.

Decay resistance of DBF-treated and DDAC-treated wood specimens

The mass losses in wood specimens treated with DBF and DDAC and exposed to the brown-rot fungus, *Fomitopsis palustris*, and the white-rot fungus, *Trametes versicolor*, are shown in Figs. 2 and 3, respectively.

The present results of the decay resistance test indicated no remarkable difference in effectiveness between DBF and DDAC regardless of weathering. Previous studies have suggested that the toxic threshold values that prevent termite and fungal attack varied with wood species.[4,13] This was true for the results in the current research. The combined data of the previous and current experiments additionally

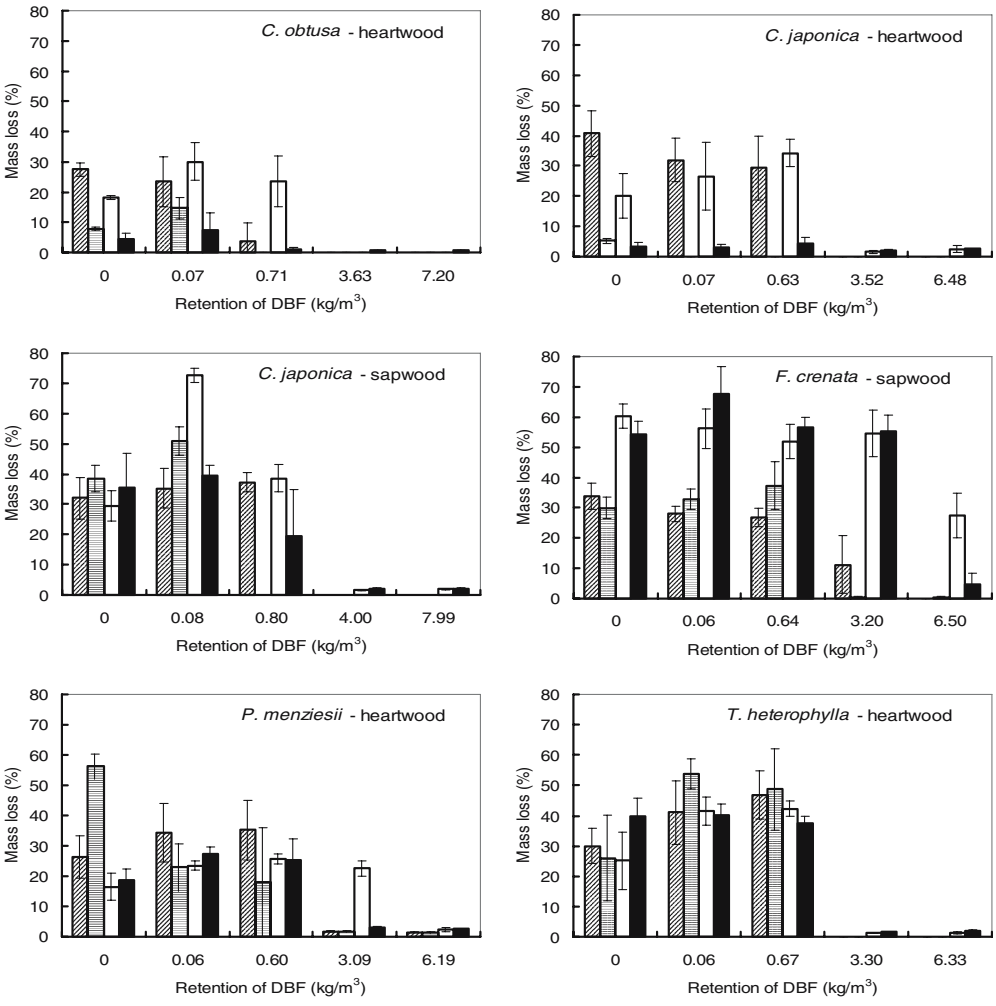

Fig. 2. Mass losses of DBF-treated and untreated wood specimens at various retention levels after 12-week exposure to the brown-rot fungus *Fomitopsis palustris* and the white-rot fungus *Trametes versicolor*. *Error bars* represent the standard deviations. *Diagonally hatched bars*, *F. palustris*-weathered; *horizontally hatched bars*, *F. palustris*-unweathered; *open bars*, *T. versicolor*-weathered; filled bars; *T. versicolor*-unweathered

Table 3. Toxic thresholds of AACs determined with unweathered wood specimens in the laboratory test

Wood species		AAC	Toxic threshold (kg/m^3)			
			F. palustris	*T. versicolor*	*C. formosanus*[a]	Overall
C. japonica	Heartwood	DBF	<0.07	0.63–3.52	<0.06	0.63–3.52
		DDAC	<0.06	0.66–3.38	<0.07	0.66–3.38
C. obtusa	Heartwood	DBF	0.07–0.71	0.07–0.71	<0.07	0.07–0.71
		DDAC	0.07–0.67	0.07–0.67	<0.07	0.07–0.67
P. menziesii	Heartwood	DBF	0.60–3.09	0.060–3.09	0.06–0.60	0.60–3.09
		DDAC	0.60–3.01	3.01–6.09	0.06–0.59	3.01–6.09
T. heterophylla	Heartwood	DBF	0.67–3.30	0.67–3.30	0.63–3.03	0.67–3.30
		DDAC	0.64–3.15	0.64–3.15	0.59–2.97	0.64–3.15
C. japonica	Sapwood	DBF	0.08–0.80	0.80–4.00	0.80–3.99	0.80–4.00
		DDAC	0.80–4.00	0.80–4.00	0.79–3.99	0.80–4.00
F. crenata	Sapwood	DBF	0.64–3.20	>6.50	0.61–3.03	>6.50
		DDAC	3.19–6.34	3.19–6.34	0.06–0.61	3.19–6.34

[a] Figures are based on the previous experimental data[4]

indicate that there is an effect of weathering on the biological performance, as shown in Tables 2 and 3. These tables summarize the toxic thresholds of DBF and DDAC against two decay fungal species and a single subterranean termite species in the laboratory before and after weathering. Some earlier works have demonstrated that a few factors such as wood structure, the chemical compositions of the tested preservatives, and natural durability including heartwood extractives played important roles in the fixation and leaching of preservatives.[14–17]

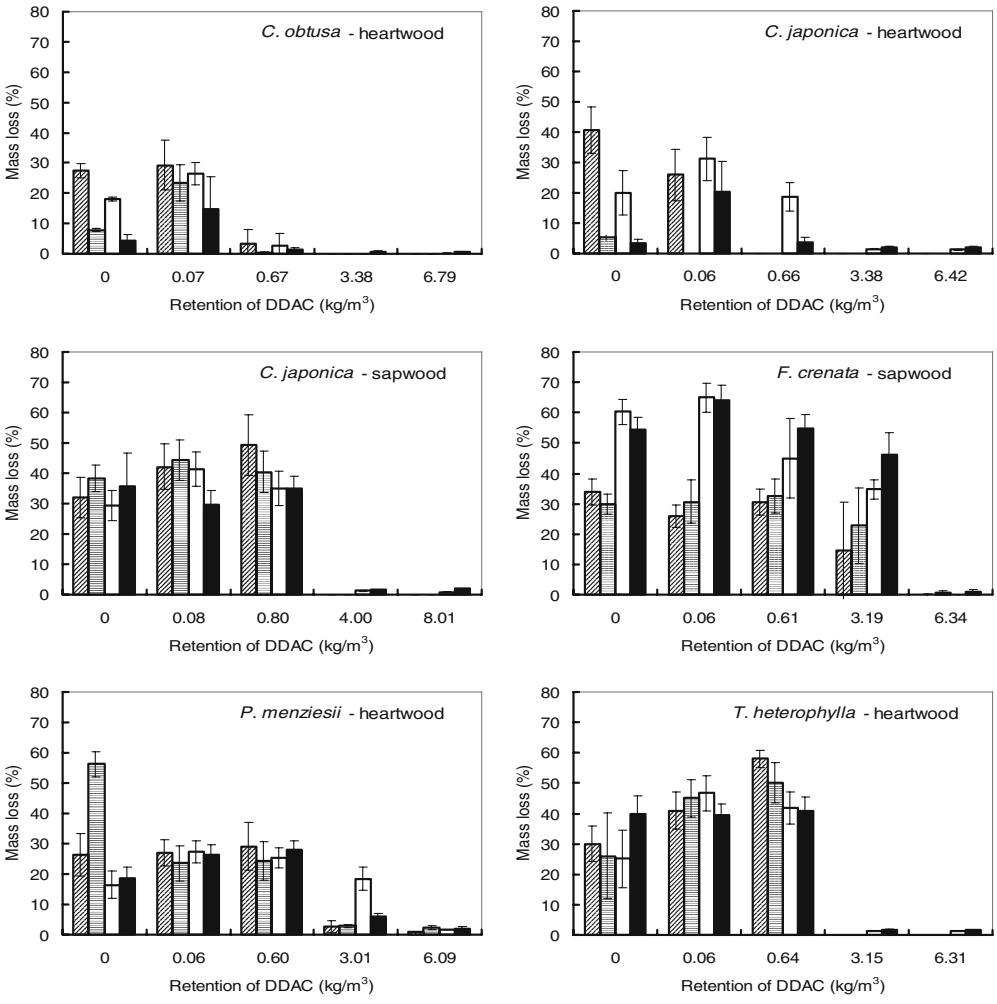

Fig. 3. Mass losses of DDAC-treated and untreated wood specimens at various retention levels after 12-week exposure to the brown-rot fungus *Fomitopsis palustris* and the white-rot fungus *Trametes versicolor*

Table 4. Efficiency of didecyldimethylammonium tetrafluoroborate (DBF) and didecyldimethylammonium chloride (DDAC) treatments in inhibiting the growth of mold and sapstain fungi on the sapwood of *Cryptomeria japonica* and *Fagus crenata*

AAC	Wood species	Treating conc. (%)	Average rating score[a]					Total of rating score	Degree of damage
			T1	T2	T3	T4	T5		
DBF	*C. japonica* (sapwood)	0.01	3.0	3.0	3.0	2.0	3.0	14.0	93
		0.10	1.3	2.0	2.0	2.0	2.2	9.5	63
		0.50	1.2	2.0	1.2	0.7	0.0	5.0	33
		1.00	0.0	1.8	0.0	0.7	0.3	2.8	19
	F. crenata (sapwood)	0.01	3.0	3.0	3.0	1.3	3.0	13.3	89
		0.10	3.0	2.8	3.0	0.5	2.7	12.0	80
		0.50	1.7	2.2	2.0	0.0	0.3	6.2	41
		1.00	1.2	2.0	0.0	0.0	0.0	3.2	21
DDAC	*C. japonica* (sapwood)	0.01	2.8	3.0	2.3	3.0	3.0	14.2	94
		0.10	3.0	2.5	2.0	1.2	0.3	9.0	60
		0.50	2.2	2.0	2.0	0.0	1.0	7.2	48
		1.00	0.0	2.0	2.2	0.0	0.7	4.8	32
	F. crenata (sapwood)	0.01	3.0	3.0	3.0	2.5	3.0	14.5	97
		0.10	3.0	2.7	3.0	1.0	3.0	12.7	84
		0.50	3.0	2.8	3.0	0.0	0.5	9.3	62
		1.00	3.0	2.0	2.0	0.0	0.2	7.2	48
Control	*C. japonica* (sapwood)	0.00	3.0	3.0	3.0	3.0	3.0	15.0	
	F. crenata (sapwood)	0.00	3.0	3.0	3.0	3.0	3.0	15.0	

[a]Test fungi T1: *Aspergillus niger*, T2: *Aureobasidium pullulans*, T3: *Gliocladium virens*, T4: *Rhizopus stalonifer*, T5: *Penicillium funiculosum*

Mold and sapstain resistance of DBF-treated and DDAC-treated wood specimens

Because the treatment solution uptake by each wood specimen in the dip-treatment was thought to be much different between the two wood species tested, the same amount of solution was applied to minimize the effect of variations in solution uptake among wood specimens.[8] The efficacy of DBF and DDAC in inhibiting the growth of mold and sapstain fungi at varying concentrations is given in Table 4.

The untreated *C. japonica* and *F. crenata* sapwood specimens were fully covered with fungal spores by the end of the second week after incubation. Dose dependence was clearly seen in the two AACs regardless of wood species. The effect of wood species is not remarkable, although *F. crenata* seemed to be more susceptible than *C. japonica* to mold and sapstain fungi when degrees of damage were compared.

In addition, no conspicuous discrepancy in effectiveness was seen between DBF and DDAC. However, a comparison of treatment concentrations, that could suppress the fungal growth at mean rating scores of less than 2.0 against the five test fungi, indicated the superiority of DBF over DDAC. Treatment could be considered efficacious in only limited cases: in ≥0.5% and ≥1.0% DBF for *C. japonica* and *F. crenata*, respectively, and in none of the DDAC concentrations for any wood species.

Performance requirements for antimold/sapstain chemical formulations have not yet been established because seasonal variations in microbial flora, the effect of wood species, the method of storage of the treated material, as well as other factors greatly influence the activity of microbial attack. Although it is therefore difficult to draw conclusions concerning the toxic limits of DBF and DDAC, the treatment with DBF at 0.5%–1.0% appears to be practically effective, assuming that the degree of damage of less than 40 without the rating score higher than 2.0 against any test fungus, is a good index as an antimold and antisapstain formulation based on the data obtained with the past commercial product.[18]

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