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Evaluation of Alkylammonium Compounds for Use as Low Toxicity Wood Preservatives in Japan*

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Abstract——Didecyl dimethyl ammonium chloride, an alkylammonium compound (AAC) was tested for potential as a wood preservative using the method prescribed by Japanese Industrial Standard JIS A 9302 and by a technique developed at the New Zealand Forest Research Institute (NZFRI) for determining effectiveness of preservatives in protecting timber used in above-ground situations. Amendments to AAC treating solutions by addition of cupric chloride, which in practice may be required to increase activity against soft rot fungi, and ammonium hydroxide were evaluated to determine their effect on AAC activity against brown and white rot fungi. *Cryptomeria japonica* and *Pinus radiata* were used as test substrates and the effectiveness of the AAC solutions was compared with that of a proprietary copper-chrome-arsenate (CCA) preservative.

At equivalent retentions, AAC was more effective than CCA in controlling decay in the JIS A 9302 test, but was less effective in the NZFRI test. Copper chloride and ammonium hydroxide amendments improved AAC effectiveness against white rot, but in dilute solutions adversely affected activity against brown rot. Differences in basic densities of the two test timbers resulted in differences in preservative retentions, particularly when these were expressed as a percentage weight/weight ratio, and some disparity in preservative effectiveness between each substrate was recorded.

Introduction

There is increasing awareness in many countries of the need for alternative chemicals for the protection of wood against decay and insect attack. This is mainly due to pressures brought by Governments and Environmental Agencies on currently used preservatives such as CCA and chlorinated phenols because of their potentially detrimental effects on health and the environment. Several reviews of the problems involved in developing new wood preservatives have been given[1~3] as well as results towards this goal[4,5].

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Of all avenues explored, the quaternary ammonium or alkylammonium compounds offer one possible solution which at present may be the most realistically acceptable. AAC have many advantages over other candidate chemicals. In addition to possessing generally low mammalian toxicity, they are freely available being used extensively in household and industrial disinfectants, in fabric softeners and conditioners and in anti-static agents.

Much work has been done in New Zealand[6~10] and elsewhere[3,11] on determining the effectiveness of many examples of AAC in controlling decay and insect attack of wood. In New Zealand, this has resulted in the approval of some AAC (benzalkonium chlorides and tertiary amine acetates) by the Timber Preservation Authority for the treatment of wood which will be used in above-ground situations[12]. Such approval, however, is restricted to the treatment of *Pinus radiata* D. Don.

Approval has been based largely on exhaustive laboratory tests in which the efficacies of various AAC have been compared with those of other chemicals whose effectiveness in service has been established for many years. Extensive field and fungus cellar tests, however, have revealed some deficiencies in AAC for the protection of wood in ground contact[13]. These deficiencies have been indicated by superficial degrade of test stakes treated to retentions of AAC which laboratory tests suggested would afford adequate protection against decay. However, it has been shown that this degrade can be controlled by addition of copper salts to treating solutions, but such amendment may not necessarily be the ideal answer to the problem.

Little work has been published in Japan on the potential of AAC as wood preservatives. A preliminary investigation[14] indicated that a tertiary amine acetate tested according to Japanese Industrial Standard procedures[15] would be unacceptable because of its lack of resistance to weathering in the designated test timber (*Cryptomeria japonica* D. Don). This result was in marked contrast to those obtained at NZFRI using *P. radiata* as test timber which showed tertiary amine acetates to be superior to many AAC in laboratory basidiomycete decay tests[8,9], although subsequent field tests have shown tertiary amine acetates to be inadequate in controlling soft rot[1].

Recent work at NZFRI has indicated that of AAC so far evaluated, the dialkyl dimethyl type may have greatest potential as wood preservatives. As a basis for developmental work on AAC wood preservatives in Japan, it was considered that a representative of this type of AAC would be a suitable choice to test under laboratory conditions prescribed in JIS A 9302. Because timber species can strongly influence wood preservative efficacy, it was thought desirable to include two wood species in the tests, *C. japonica* and *P. radiata,* the latter being the softwood species in which most AAC evaluation work has been undertaken in New Zealand.

Because of doubts about the resistance to weathering of AAC in *C. japonica,* modifi-

cations were made to preservative solutions to try to improve fixation. These mainly involved the use of ammoniacal AAC solutions. Preservative solutions were also amended by addition of cupric chloride and cuprammonium chloride. This was done mainly because field tests to determine the resistance of AAC-treated stakes to natural populations of decay fungi were to be subsequently established and such amendment would be necessary to control soft rot. It was considered desirable to determine whether such amendments affected activity of AAC against basidiomycete decay in laboratory tests.

Any approval for use of AAC as a wood preservative in Japan will most likely be restricted initially to timber which will be used only in moderate decay hazard situations. Therefore it was considered appropriate to carry out concurrent tests under conditions which simulated such hazards using preservative formulations similar to those used in JIS A 9302 tests. The test procedure adopted was that developed at NZFRI[16].

Materials and Methods

A. Preservative solutions

Stock solutions of the following chemicals were prepared;

(a) 1.25% Celcure K33, a copper-chrome-arsenate preservative with the following composition: CuO: 14.8%, CrO_3: 24.6%, As_2O_5: 34.0%, H_2O: 24.6%
(b) 1.0% active ingredient (a.i.) Bardac 2250, didecyl dimethyl ammonium chloride
(c) 1.0% a.i. Bardac 2250+0.45% NH_4OH
(d) 1.0% a.i. Bard0c 2250+0.50% $CuCl_2 \cdot 2H_2O$
(e) 1.0% a.i. Bardac 2250+0.50% $CuCl_2 \cdot 2H_2O$+0.45% NH_4OH
(f) 1.0% a.i. Bardac 2250+0.25% $CuCl_2 \cdot 2H_2O$
(g) 1.0% a.i. Bardac 2250+0.25% $CuCl_2 \cdot 2H_2O$+0.45% NH_4OH

B. JIS A 9302 Test

Each solution was used at 100%, 50% and 25% of its stock solution concentration. Twenty-four blocks each of *C. japonica* and *P. radiata* sapwood measuring 20×20×10 mm, the grain running parallel to the shorter dimension, were treated to saturation with each solution by vacuum/soak impregnation (30 min vacuum, 2 h soak). Blocks were weighed oven dry before treatment and in a saturated condition immediately after treatment, from which preservative retentions in each block were calculated. Following treatment, blocks were wrapped in polyethylene and stored for two weeks to allow for preservative fixation. Blocks were then air-dried and 12 of each retention and timber species were weathered as prescribed in JIS A 9302 (blocks placed in running water flowing at a rate of 1–2 *l*/min for 1 h, oven dried at 60°C for 22 h and the cycle repeated 9 times).

Decay vessels were 800 ml capacity round glass jars containing 250 g quartz sand moistened with 80–85 ml nutrient solution containing 2.5% glucose, 0.5% peptone 1.0% malt extract, 0.3% KH_2PO_4 and 0.2% $MgSO_4 \cdot 7H_2O$. Jars were sterilised by autoclaving and inoculated with either *Tyromyces palustris* (Berk. ex Curt.) Murr. FES 0507* (brown rot) or *Coriolus versicolor* (L. ex Fr.) Quél. FES 1030* (white rot).

All blocks, including untreated controls, weathered and unweathered were oven dried, weighed and sterilised by exposure to ethylene oxide gas. Three blocks of the same timber species and preservative retention were aseptically placed in each jar, equal numbers of blocks being exposed to each fungus. Jars were incubated at 27°C for 90 days. Following incubation, blocks were cleaned of adherent mycelium, oven dried, weighed and percentage weight losses determined.

C. NZFRI Above-ground Test

Treating solutions were 20% and 10% dilutions in distilled water of stock solutions. Thirty-two blocks of each timber species were treated with each solution using the same procedure as in the JIS A 9302 test. After fixation and air-drying, equal numbers of blocks were weathered according to JIS A 9302 and by standard NZFRI methods (blocks impregnated with water, placed in nine times their own volume of water which was changed every two days for 14 days). Following weathering, blocks were oven dried, weighed and sterilised by exposure to ethylene oxide gas.

Square glass jars (1500 ml capacity) containing 2% malt extract agar were prepared so that the agar covered one side of each jar when placed horizontally. Jars were inoculated with either *T. palustris* or *C. versicolor*. Feeder blocks measuring 15 × 20 × 160 mm were prepared from *C. japonica* sapwood. Nine slots, 12 mm wide × 10 mm deep were cut into a 20 × 160 mm face of each block before sterilisation with ethylene oxide gas. Two feeder blocks were placed in each jar, supported above the inoculated agar surface by rests constructed from 3.0 mm glass rod. Jars were incubated at 27°C for 28 days, by which time feeder blocks were completely overgrown and heavily infected with the decay fungi. Feeder blocks were then aseptically transferred to similar glass jars containing water agar. Two feeder blocks supported by glass rests were placed in each jar. Sterilised test blocks were then inserted in the slots of each feeder block so that each feeder block held 9 different treatments of a single timber species and the two feeder blocks in each jar held an identical range of treatments but of different timber species. Jars were then incubated at 27°C for 90 days. Following incubation, percentage weight losses were determined for each block after cleaning and oven-drying.

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Results

1. JIS A 9302 Test

Results are given in Table 1 and are expressed in the format required by JIS A 9302. " Values of Efficiency " of each preservative shown in Table 1 were derived from the equation:

$$(W_1-W_2)/W_1\times 100$$

where, W_1 = Mean percentage weight loss of untreated blocks.

W_2 = Mean percentage weight loss of treated blocks.

Weight losses of untreated *P. radiata* blocks decayed by *T. palustris* which are shown in Table 1, were lower than normally encountered for perishable timbers exposed to this fungus in the JIS A 9302 test. Low weight losses in control blocks tend to reduce calculated Values of Efficiency and therefore there is a possibility that those shown in Table 1 are lower than would normally be derived from the JIS A 9302 test using *T. palustris* as test fungus. For this reason, more emphasis has been placed on results with *C. versicolor* to illustrate differences in performance of the various preservatives in the two timber species.

At equivalent treating solution concentrations, effectiveness of all preservatives was greater in *C. japonica* than in *P. radiata,* even after allowing for the slightly higher preservative retentions weight/volume in the former. However, because of large differences in wood basic densities of the two species, preservative retentions when expressed as a percentage weight/weight ratio for any one solution concentration differed markedly between species as indicated in Table 1.

The only significant weight losses of treated *C. japonica* blocks exposed to *T. palustris* were in weathered blocks containing 2.5 kg CCA/m^3. *C. versicolor* caused slightly more decay than *T. palustris* in CCA-treated *C. japonica* blocks, but insignificant amounts in AAC-treated blocks. The results indicate that in *C. japonica* the toxic threshold (defined as the minimum preservative retention which prevents decay) of didecyl dimethyl ammonium chloride, whether or not amended, was below 2–3 kg/cm^3 against either fungus.

Results with *P. radiata* showed clearer differences in effectiveness of the various solutions. Weathering had only slight effect on the efficacy of CCA. *C. versicolor* caused significant weight losses in blocks treated to 9.0 kg/m^3 with CCA, but *T. palustris* was less tolerant to this preservative.

Didecyl dimethyl ammonium chloride was more effective than CCA in controlling decay by both fungi, the toxic threshold being less than 3.3 kg/m^3. At the lowest retention tested, weathering substantially reduced effectiveness against *C. versicolor,* but had negligible effect on decay by *T. palustris.* Effectiveness of AAC

Table 1. Effectiveness of AAC preservative solutions in JIS A 9302 test.

Treating solutions		Pinus radiata						Cryptomerria japonica					
		Retention		Value of Efficiency				Retention		Value of Efficiency			
				T. palustris weathered		C. versicolor weathered				T. palustris weathered		C. versicolor weathered	
		kg/m³	% w/w	Yes	No	Yes	No	kg/m³	% w/w	Yes	No	Yes	No
CCA (Celcure K33)	1	9.0	2.34	95	100	84	94	10.0	3.84	100	100	98	98
	2	4.5	1.09	82	74	63	71	5.0	1.91	100	100	92	96
	3	2.3	0.56	—*	66	67	42	2.5	0.97	89	99	76	94
AAC (Bardac 2250)	1	7.2	1.79	99	98	100	100	8.0	3.18	100	100	100	100
	2	3.3	0.69	97	94	100	97	4.0	1.50	100	100	100	100
	3	1.7	0.35	73	89	65	88	2.0	0.77	100	100	100	100
AAC+NH_4OH	1	6.8	1.42	96	93	99	97	8.0	2.91	100	100	100	100
	2	3.4	0.70	92	93	98	97	4.0	1.44	100	100	100	99
	3	1.8	0.43	82	98	80	93	2.0	0.75	100	100	100	97
AAC+$CuCl_2 \cdot 2H_2O$ 2 : 1	1	10.8	2.66	100	94	100	99	11.7	4.35	100	100	100	100
	2	5.0	1.02	95	95	97	97	6.0	2.22	100	100	100	99
	3	2.5	0.52	—*	87	88	95	3.0	1.16	100	100	99	100
AAC+$CuCl_2 \cdot 2H_2O$+NH_4OH 2 : 1	1	10.7	2.64	100	100	99	98	11.9	4.56	100	100	100	99
	2	5.0	1.04	90	97	98	94	5.9	2.19	100	100	98	98
	3	2.5	0.53	—*	94	78	93	2.9	1.12	100	100	100	99
AAC+$CuCl_2 \cdot 2H_2O$ 4 : 1	1	8.7	2.08	96	90	98	96	9.9	3.86	100	100	100	100
	2	4.2	0.87	97	88	98	96	4.9	1.91	100	100	100	100
	3	2.1	0.43	—*	90	85	97	2.5	0.96	100	100	100	100
AAC+$CuCl_2 \cdot 2H_2O$+NH_4OH 4 : 1	1	8.9	2.22	100	100	100	97	10.0	3.91	100	100	100	100
	2	4.2	0.87	98	96	98	96	4.9	1.78	100	100	100	100
	3	2.1	0.45	84	94	87	84	2.5	0.99	100	100	97	99
Percentage weight weight losses of untreated controls				9.6	17.9	48.2	42.4			37.6	15.2	38.6	49.0

Note: Solutions 1, 2, 3 correspond to 100, 50 and 25% solutions respectively of original stock solution.
* weight losses of treated blocks were higher than those of untreated blocks.

increased slightly in ammoniacal solution. Addition of cupric chloride to treating solutions also increased effectiveness against *C. versicolor,* but at the lower retention tested had a reverse effect against *T. palustris* particularly in weathered specimens. This pattern was repeated when cuprammonium chloride was used instead of cupric chloride. Reducing the proportion of Cu in treating solutions had little effect on preservative efficacy against *C. versicolor,* but apparently increased effectiveness against *T. palustris.*

2. *NZFRI Above-ground Test*

Results are shown in Table 2 in which mean percentage weight losses of test specimens are used to indicate the effectiveness of preservative solutions. As in the JIS A 9302 test, performance of all solutions was better in *C. japonica* than in *P. radiata,* presumably for the same reasons as outlined above. CCA gave slightly greater protection to test blocks than unamended AAC. Addition of NH_4OH to AAC reduced decay in blocks treated to the higher retention and exposed to *C. versicolor,* but otherwise it had little effect. Amending AAC solutions with copper also only significantly increased effectiveness against the white rot fungus. With *T. palustris,* lower retentions of copper tended to stimulate decay. The enhancement effect of copper

Table 2. Effectiveness of AAC preservative solutions in NZFRI Above-ground test.

Treating solutions		Pinus radiata					Cryptomeria japonica				
		Retn kg/m³	T. palustris % weight loss weathering		C. versicolor % weight loss weathering		Retn kg/m³	T. palustris % weight loss weathering		C. versicolor % weight loss weathering	
			JIS	FRI	JIS	FRI		JIS	FRI	JIS	FRI
CCA (Celcure K33)	4	1.6	5.4	5.2	1.3	1.1	2.0	1.6	1.7	0.8	0.7
	5	0.8	11.0	10.7	1.4	0.5	1.0	5.9	5.4	1.4	0.9
AAC (Bardac 22)	4	1.3	11.7	6.4	9.9	7.0	1.6	15.3	4.4	4.2	4.7
	5	0.6	22.3	12.5	11.7	12.7	0.8	22.8	17.5	8.2	9.1
AAC+NH_4OH	4	1.3	15.1	4.1	1.7	1.8	1.6	1.7	2.3	1.7	1.5
	5	0.6	24.7	20.5	10.2	10.0	0.8	23.9	4.1	9.5	6.7
AAC+$CuCl_2 \cdot 2H_2O$ 2 : 1	4	1.9	11.1	8.8	1.2	1.2	2.3	5.0	2.4	0.7	1.3
	5	1.0	17.4	13.9	1.3	1.2	1.2	9.5	3.3	1.0	1.2
AAC+$CuCl_2 \cdot 2H_2O$ 2 : 1 +NH_4OH	4	1.9	12.3	3.9	2.2	0.8	2.2	1.4	1.0	0.8	0.9
	5	0.9	34.7	27.2	9.7	9.1	1.1	12.3	6.4	7.9	6.6
AAC+$CuCl_2 \cdot 2H_2O$ 4 : 1	4	1.6	8.3	5.3	1.4	1.7	1.9	3.8	1.8	1.2	1.5
	5	0.8	24.2	11.3	2.1	1.8	1.0	9.6	6.6	0.7	1.0
AAC+$CuCl_2 \cdot 2H_2O$ 4 : 1 +NH_4OH	4	1.6	5.9	2.7	1.8	1.4	2.0	3.9	0.5	0.5	1.4
	5	0.8	34.7	15.3	8.7	6.2	1.0	12.9	5.0	8.7	7.7
Not treated (not weathered)			46.7		23.7			50.6		15.6	

Note: Solutions 4, 5 correspond to 20 and 10% dilutions respectively of original stock solutions.

on total preservative efficacy against *C. versicolor* was negated if cuprammonium rather than cupric ions were used. Reducing Cu ion proportions in AAC solutions had negligible effect on activity in all instances.

Discussion

The results demonstrate that didecyl dimethyl ammonium chloride is more effective than CCA in controling decay by brown and white rot fungi in the severe decay hazard environment of the JIS A 9302 test. According to JIS A 9201 (Qualitative standards for wood preservatives), Grade A preservatives, which if they are to be approved for general use, require Values of Efficiency of at least 90 against *T. palustris* and at least 80 against *C. versicolor*. The only CCA retentions which passed these criteria were 9.0 kg/m^3 in *P. radiata* and between 2.5 and 5.0 kg/m^3 in *C. japonica*. In comparison, unamended AAC at a retention of between 1.7 and 3.3 kg/m^3 in *P. radiata* and 2.0 kg/m^3 in *C. japonica* would qualify as A Grade preservatives.

Amending treating solutions with ammonia, cupric ions or cuprammonium ions gave variable results and the value of these additives in improving preservative performance against brown and white rot was not conclusively demonstrated. From results with *C. japonica*, it was not possible to determine whether or not fixation had been improved by addition of ammonia, but in any case this would appear to be unnecessary since unamended solutions were equally effective as amended ones. Ammoniacal AAC treatment of *P. radiata* was more effective than aqueous AAC treatment, particularly against *C. versicolor*. However, this improvement may not be advantageous in practice. AAC in alkaline solutions fixes more rapidly to wood than when in neutral or acid solution[6]. Consequently, in the present work, this could have resulted in higher concentrations of AAC preventing attack in outer regions of test blocks at the expense of lower, sub-toxic, retentions in the centres of blocks.

Addition of cupric chloride to treating solutions had negligible effect on activity of AAC to *C. versicolor*. However, it substantially reduced effectiveness against *T. palustris* in weathered blocks treated to low retentions with a 2 : 1 mixture of AAC to $CuCl_2 \cdot 2H_2O$. This suggests that the copper concentration after weathering was optimal for stimulation of growth and activity of the fungus; a phenomenon not unusual in other decay fungi exposed to sub-lethal doses of toxic ions[17,18]. In practical terms, this result may be of little importance because such dilute solutions would not be used for commercial timber treatments. However, the result does indicate that amendment of AAC-treating solutions to improve performance against, for example, soft rot fungi, may have a detrimental effect on activity against other fungi.

In the above-ground test, toxic values for CCA (which may be defined as the

maximum retention of preservative which allows decay and the minimum retention which prevents decay) in *C. japonica* were 1.0–2.0 kg/m^3 *(T. palustris)*, $<$1.0 kg/m^3 *(C. versicolor)* and $>$1.6 kg/m^3 *(T. palustris)*, $<$0.8 kg/m^3 *(C. versicolor)* in *P. radiata*. In this test, unamended AAC was less effective than CCA and results indicated poorer performance of AAC in *C. japonica* than in *P. radiata*. Although weight losses of test blocks were too high for accurate computation of toxic values, extrapolation of weight loss figures suggests toxic values in the order of 2.0–2.5 kg/m^3 for NZFRI-weathered test blocks of both timber species, but rather higher for *C. japonica* weathered by the JIS A 9302 method. Amendment of treating solutions with ammonia or cupric chloride did not radically improve AAC effectiveness in *P. radiata* against *T. palustris*, but substantially increased efficacy against *C. versicolor*, either at one preservative retention (ammonia amendment) or both (cupric chloride amendment). Improvement in performance in *C. japonica* against *T. palustris* was also obtained at higher retentions by ammonia or copper ion amendment. On the other hand, cuprammonium ions had an adverse effect on performance particularly at lower retentions in *P. radiata* exposed *T. palustris* after JIS A 9302 weathering.

The NZFRI above-ground test was originally developed to test preservative efficiency under conditions which by restricting nutrient and moisture supply, were sub-optimal for fungal growth, hence simulating a situation which would occur in timber used in above-ground situations in constructions (*e.g.* house sills). In previous tests, toxic values of preservatives have been found to be lower than in soil/block tests (which simulate ground-contact conditions) and AAC has been shown to be superior to CCA when *Gloeophyllum trabeum* (Pers. ex Fr.) Murr. (brown rot) has been used as test fungus[16].

Such results have been used as a basis for approval of AAC for use at lower retentions than CCA in aboveground situations[12]. The superiority of AAC over CCA was not apparent in the present tests, which may reflect differences in weathering procedures and differences in fungus species used. Thus, if this type of test was accepted in Japan as a basis for preservative approval for above-ground use, it would be difficult to justify the use of AAC at retentions lower than for CCA.

Conclusions

The effectiveness of didecyl dimethyl ammonium chloride (AAC) in controling decay by brown and white rot fungi was demonstrated in JIS A 9302 tests in which performance of AAC was superior to that of a copperchrome-arsenate (CCA) preservative. Weathering of treated test blocks before exposure to the decay fungi reduced the effectiveness of AAC at the lowest retentions tested (1.7 $\sim$2.5 kg/m^3) and reduction in effectiveness against brown rot was pronounced when treating solutions

were amended with cupric or cuprammonium ions. When tested in more dilute solutions in a lower decay hazard environment, AAC was inferior to CCA and there was a greater reduction in effectiveness against brown rot following addition of copper to treating solutions.

The results indicate that within the range of solution concentrations tested, there was only a gradual change in effectiveness of CCA as preservative retentions increased or decreased, whereas with AAC an abrupt change in effectiveness took place over a narrow retention range, the degree of change being strongly influenced by amendments to treating solutions. It will be necessary to recognise this feature of AAC during further evaluation of this group of chemicals as low toxicity wood preservatives.

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