

AMMONIACAL COPPER/QUATERNARY AMMONIUM COMPOUND WOOD PRESERVATIVE SYSTEMS

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Abstract

With the growing environmental pressures on conventional wood preservatives there has been increasing interest in North America and elsewhere in the development of new systems of lower mammalian toxicity that would present less potential for environmental impact and would allow for easier disposal of treated products withdrawn from service. Ammoniacal copper/quat (ACQ) preservatives have been developed to meet these objectives. Some of the work leading to identification of the preferred systems of this type is described here. The areas covered include early screening work, fungus cellar studies, field and marine tests and plant trials. The results show that, for the preferred systems, treating characteristics, performance in ground-contact and performance in marine applications compare favourable with those of currently used preservatives.

Background

Quaternary ammonium compounds are produced by alkylation of amines, and those of biocidal interest are water soluble salts. They are well-established biocides and are approved for use in a variety of applications such as for disinfection of surfaces in schools, hospitals, dairy facilities, etc. Their low mammalian toxicity is apparent from these uses as well as from their approval for use on food processing equipment and in laundry applications. Quats are also approved in North America for use in wood protection and in wood preservation where their substantivity to wood substance provides them with a fixation mechanism.

The potential of quats as wood preservatives has been studied extensively (2, 4, 9, 10, 15) and it has been shown that quats can protect wood in above-ground applications. However, when used alone, they do not provide sufficient protection for wood in ground contact (11, 15). Work on amended quat systems showed that performance is improved by addition of metals such as copper (3, 4, 5, 15, 16). Early systems of this type were based on water-soluble copper salts, such as the chloride or the sulphate, and typically involved combinations in which quat was the main component and the relative amount of copper was low. In field tests, systems of this type did not provide the desired level of protection (12, 15).

In contrast to the above findings, ammoniacal copper/quat systems which are based on insoluble copper and formulated with higher copper to quat ratios were found to

have the potential for use in ground contact applications (6, 14, 17, 13). Recent publications on this type of system have covered such aspects as dimensional stability, fixation and preservative distribution (7, 8).

Introduction

The approach taken by Domtar in development of ACQ systems was to use copper as the primary biocide, with quats being used as co-biocides to control copper-tolerant organisms. Use of an ammoniacal carrier allowed a water-insoluble form of copper, copper carbonate, to be used in the formulations. After impregnation into wood and subsequent drying, the copper becomes fixed as insoluble copper carbonate and through ion-exchange to wood substance. The quaternary ammonium compound fixes due to the affinity between it and wood substance. Being ammoniacal systems, ACQ preservatives are particularly suitable for treatment of refractory Canadian wood species that are difficult to treat with non-ammoniacal water borne preservatives.

Materials

ACQ treating solutions were formulated from ammoniacal copper carbonate concentrates prepared either directly from cupric carbonate or else by air-oxidation of cuprous oxide in ammonium hydroxide containing carbonate anion. On a weight basis, the amount of carbonate (expressed as CO_2) in each system was 0.66 times the amount of copper (expressed as CuO). The amount of ammonia in all systems was about 1.8 times the amount of CuO .

The quats used in the systems described here, and the acronyms used in this paper are the following:

ODAC octyldecyldimethylammonium chloride ($\text{C}_8,10$ 50%, $\text{C}_8,8$ 25%, $\text{C}_{10,10}$ 25%)
DDAC didecyldimethylammonium chloride ($\text{C}_{10,10}$ 100%)
ABAC alkylbenzyldimethylammonium chloride (C_{12} 40%, C_{14} 50%, C_{16} 10%)

The ODAC and DDAC were Bardac 20 and Bardac 22 respectively, obtained from Lonza. The ABAC was obtained from Matheson, Coleman and Bell. Ratios shown for particular ACQ systems refer to the ratio by weight of copper oxide to quaternary ammonium compound.

Initial Screening for Biologically Effective Systems

Individual quats differ in terms of fungicidal effectiveness, and preliminary

studies involved screening quats in combination with copper to identify promising candidate systems. The screening method used in this early work was that of Bravery (1) in which small treated blocks (5 x 10 x 20 mm) are placed on polyethylene screens over fungi growing on agar plates. All blocks were leached prior to exposure to the test organisms. The data in Table 1 show the results for a number of systems based on ammoniacal copper and quat, in which the copper oxide to quat ratio was 2:1. The systems tested were found to be equally effective against *G. trabeum* and *L. lepideus*. Against copper-tolerant *P. placenta*, however, performance is seen to be dependent on the particular quat used. Against this organism, all systems containing quat were superior to copper carbonate alone. In other screening work, alternative systems incorporating other fungicidal anions such as borate and fluoride were tested, but were not found to be more effective than systems based on copper and the corresponding quat without the additional anion.

The effect of varying the copper to quat ratio was also investigated. The results for systems based on ODAC, and leached prior to testing, are shown in Table 2. For *P. placenta*, improved performance of systems having copper oxide to quat ratios ranging from 5:1 to 1:5 can be seen relative to the performance of copper carbonate when used alone. The results for ABAC systems show a similar pattern of results.

This early work showed certain quats to be more effective than others and indicated further that ammoniacal systems containing copper and quat were effective over a range of copper to quat ratios. These findings were borne out by the results of standard soil block tests and were further substantiated by the results of fungus cellar tests.

Fungus Cellar Studies

Red pine and white birch stakes (9.5 x 12.5 x 200 mm) were full-cell treated with ACQ systems. After treatment, each batch of ten replicate stakes was stored under a polyethylene shroud for one week prior to air-drying. All material was leached for three days under running tap water, then air-dried, prior to installation in the fungus cellar. The soil beds contained agricultural soil with a water holding capacity of 29%, a pH of 5.2, and an organic matter content of 5.8%. Relative humidity and temperature were maintained at 70-90% and 27-31°C respectively. During the exposure period, soil moisture was maintained at 60-80% of water holding capacity. Table 3 shows the results for systems formulated with ODAC after exposure for twelve months, and shows that the controls had essentially all failed early in the test. Performance of the high copper content 5:1 and 2:1 systems was equal to or better than ACA at similar retentions, and slightly better than that of the 1:5 system. The data for birch stakes treated with the 2:1 system show its effectiveness in protection of hardwood. A detailed report on the fungal cellar work was published in 1986 (17).

Field Stake Tests

Field stake tests were contracted out to Forintek Canada Corp., and to Mississippi State University. Standard test procedures (ASTM D-1758-74 or AWWA M7-83) were used, using 3/4" stakes. By this time, laboratory tests had identified dialkyldimethyl quats with alkyl chain lengths of C₈ to C₁₀ as being among the most effective when combined with copper. In Canada, where the first test stakes were installed, the systems tested were formulated with the ODAC quaternary and the test locations were the Petawawa and Ottawa test sites.

Table 4 shows the results of seven years of exposure at Petawawa, by which time the untreated controls had failed completely. The low copper (1:5) system can be seen to be performing less well than the 2:1 or 5:1 systems, while both of these are performing similarly to ACA, other than at the lowest retentions.

At Ottawa, the performance of a 1:1 ODAC system was compared with that of ACA. Table 5 shows the results after six years exposure at this site, by which time the untreated controls had failed completely while the two test systems are performing equally well.

ODAC systems with copper oxide to quat ratios of 3:1, 1:1 and 1:3 are undergoing standard field tests at two sites in Mississippi. These have been in progress for five years. At both sites, the untreated controls failed completely at about two years. The 1:3 system is performing slightly less well at both sites than the systems with higher copper contents. At the Dorman site, the quat-based systems are performing slightly less well than CCA-C in terms of decay attack, and similarly to CCA-C in terms of termite attack. At the Saucier location, the quat-based systems of higher copper content are performing similarly to CCA-C at comparable retentions in protection against both decay and termite attack.

Systems based on DDAC and on ODAC quats, with copper oxide to quat ratios of 1:1, are also being tested at the two Mississippi sites, and have been in place for three years. In these tests, southern yellow pine was used to prepare the stakes rather than the red pine that had been used in all the above stake tests.

The results of the tests at the Dorman site are presented in Table 6. These data show that the ACQ systems based on DDAC and ODAC are performing equally well and at levels close to that of CCA-C. Even the lowest ACQ retention (3.3 kg/m³) is providing good protection against termites.

Data from the tests at the Saucier site are shown in Table 7. The results show, again, that the two ACQ systems are performing equally well and at levels very close to that of CCA-C at all but the lowest retentions.

Marine Exposure Tests

Marine exposure tests were contracted out to Mississippi State University. The test site was at Ponce Inlet, Florida. The test protocol was according to ASTM D2481-81, with panels dimensions being 19 x 76 x 460 mm. Two tests are in progress

and in both of these the untreated control panels had failed by the time of the first annual inspection. One of the tests has been under way for 3 years and involved 1:1 systems based on ODAC and on DDAC. All panels are currently sound, down to the lowest retentions used (about 9 kg/m³), as are the similarly treated CCA-C controls. Attack has occurred to give a rating of 8.6 on the creosote-treated controls at the highest creosote retention used (270 kg/m³).

Table 8 shows the results of the other marine test in which the test systems were based on ODAC and covered copper oxide to quat ratios of 3:1, 1:1 and 1:3. The results show the 1:1 system to be performing slightly better than the other two, with its performance being similar to that of CCA-C at similar retentions.

Treating Studies

Treatment studies have been conducted under pilot plant conditions and in plant trials at the Delson plant in Quebec. These studies have shown the treating characteristics of ACQ systems to be similar to those of conventional ammoniacal, copper-containing preservatives. For the plant trials, a 3% ACQ (1:1) solution based on ODAC was used for treatment of roundwood and timber. The treating temperature was about 150°F and treating pressures and schedules were within normal parameters. During treatment of some twenty-five charges in the plant trials, solution composition remained essentially in balance, with no evidence of any depletion of quat from the treating solution. No sludge formation was observed. Table 9 shows the results of some of the plant treatments. In most cases, assay retentions exceeded target values and indicate that a solution concentration of less than 3% could have been used. An unexpected result was the successful treatment of Eastern hemlock of high moisture content.

The appearance of treated wood was found to be a function of the species, with pine sapwood being generally greenish and Eastern hemlock being brown. In pines, the heartwood is also brown. Red pine pole stubs were found to gradually lose their greenish colour and to become light brown on weathering.

Current Status

A considerable body of information has now been developed on ACQ systems. This includes additional information on performance, as well as on other aspects such as fixation, depletion and ancillary properties of the treated products. A data package compiled jointly by Domtar and CSI was recently presented to the AWWA Preservatives Committees in support of two systems designated as ACQ-A and ACQ-B which are based on DDAC and have copper oxide to quat ratios of 1:1 and 2:1 respectively. Committee approval of these two systems for ground contact applications at the same retentions as CCA-C was subsequently granted at the Clearwater Beach committee meetings of AWWA in September. Systems of this type are already in commercial use in Sweden and in Japan, and application has been made to Agriculture

Canada to obtain approval for use in Canada.

Conclusions

The purpose of this paper was to give an overview of some of the work that has been done in Canada leading to identification of the preferred ACQ systems. The current body of knowledge on ACQ's indicates that the preferred systems are based on the dialkyldimethyl quats, with copper to quat ratios of 1:1 to 2:1. As with any preservative system, research work will continue, and it is anticipated that ACQ's will provide a fertile ground for further studies in the future.

The information developed thus far shows ACQ's to have considerable promise for protection of wood in ground contact and in marine applications, while providing advantages in terms of environmental considerations and ease of ultimate disposal of the treated product.

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Table 1. Threshold retentions of ammoniacal copper/quat systems formulated with CuO:quat ratios of 2:1.

Quat type and trade name	Leached Threshold retentions		
	G. trabeum (kg/m ³)	L. lepidus (kg/m ³)	P. placenta (kg/m ³)
Alkylbenzyl dimethyl:			
ABAC (C12-C16)	0.46	0.17	1.8
Barquat MX-50 (C12-C18)	0.52	0.17	4.2
Barquat OJ-50 (C12-C16)	0.46	0.42	1.0
Dialkyldimethyl:			
Bardac 20 (C8-C10)	0.46	0.18	1.2
Bardac 22 (C10)	0.66	0.18	1.4
Bardac LF (C8)	1.10	0.25	2.6
Alkyltrimethyl:			
Arquad C-50 (C12-C16)	0.46	0.40	1.4
Arquad T-50 (C16-C18)	0.44	0.52	1.2
Arquad S-50 (soya)	0.44	0.22	2.2
Diquaternary:			
Duoquat T-50 (C16-C18)	1.16	0.52	3.4
CONTROLS:			
Ammoniacal copper carbonate	0.42	0.42	>10.7
ACA	1.8	0.46	2.4

Table 2. Effect of composition on leached threshold retentions.

System Tested	Leached Threshold Retentions		
	G. trabeum (kg/m ³)	L. lepideus (kg/m ³)	P. placenta (kg/m ³)
ODAC Systems:			
CuO only	<0.13	<0.13	4.5
5:1	0.35	<0.13	1.4
2:1	0.50	0.20	1.6
1:1	0.62	0.20	0.8
1:2	0.45	<0.13	0.35
1:5	0.55	0.20	0.4
Quat only	1.30	<0.13	<0.13
ABAC Systems:			
5:1	0.50	<0.13	0.35
1:1	0.50	<0.13	0.37
1:5	0.95	<0.13	0.30
Control:			
CCA-C	1.28	0.16	2.6

Table 3. Results of fungal cellar tests on ACQ (ODAC) systems.

System Tested	Nominal Retention (kg/m ³)	Analysed Retention (kg/m ³)	Average Stake Ratings (Test Time in Months)		
			5	7	12
Red Pine Stakes:					
ACQ 1:5	13.3	11.7	10.0	10.0	10.0
	7.4	6.6	10.0	9.8	9.6
	4.2	3.5	10.0	9.2	8.4
	2.2	2.1	7.4	7.4	6.8
	1.3	1.1	3.1	3.1	0.8
ACQ 2:1	9.8	8.8	10.0	10.0	10.0
	5.3	6.1	10.0	10.0	10.0
	3.0	4.0	9.9	9.9	9.7
	1.6	2.1	7.7	7.3	6.9
	0.8	1.0	3.8	3.0	3.0
ACQ 5:1	9.8	9.3	10.0	10.0	10.0
	5.3	4.6	10.0	10.0	10.0
	2.9	3.4	9.8	9.7	9.6
	1.6	1.8	7.7	7.7	7.7
	0.8	1.1	5.5	4.5	4.1
ACA	15.8	10.7	10.0	10.0	10.0
	8.5	5.9	10.0	10.0	10.0
	4.6	2.9	10.0	10.0	10.0
	2.6	1.4	9.2	8.8	8.8
	1.4	0.8	5.5	5.1	4.3
NH ₄ OH-treated controls			1.4	1.3	0.8
Birch Stakes:					
ACQ 2:1	9.6	9.1	10.0	10.0	10.0
	5.4	5.3	10.0	10.0	10.0
	3.0	2.9	10.0	10.0	10.0
	1.6	2.4	4.2	3.1	2.0
	1.0	1.4	0.8	0.4	0.0
ACA	16.2	11.2	10.0	10.0	10.0
	8.8	6.1	10.0	10.0	10.0
	4.8	3.7	10.0	10.0	10.0
	2.7	1.8	8.8	7.2	3.4
	1.4	1.0	0.8	0.8	0.0
NH ₄ OH-treated controls			0.4	0.0	0.0

Table 4. Results on red pine stakes installed at Petawawa, Ontario

System Tested	Retentions		Average Decay Ratings (Test time in years)				
	Nominal (kg/m ³)	Assay (kg/m ³)	1	3	5	6	
ACQ 1:5	1.8	1.3	8.9	4.3	1.2	1.0	0.
	3.1	2.8	9.6	6.4	4.7	1.6	0.
	5.9	2.8	10.0	8.9	7.3	8.7	6.
	10.1	8.3	10.0	9.9	9.5	10.0	9.
	19.0	11.1	10.0	10.0	10.0	10.0	10.
ACQ 2:1	1.9	1.9	8.8	7.2	4.1	2.9	2.
	3.3	3.1	9.7	9.1	6.7	5.9	4.
	6.1	5.0	10.0	9.5	9.3	9.7	9.
	11.8	10.6	10.0	10.0	10.0	10.0	9.
	20.9	18.2	10.0	10.0	10.0	10.0	9.
ACQ 5:1	2.9	2.8	10.0	8.4	7.7	6.7	6.
	5.4	4.7	10.0	10.0	9.7	9.7	8.
	9.5	9.8	10.0	10.0	10.0	10.0	10.
	17.6	13.5	10.0	10.0	10.0	10.0	10.
	33.5	30.2	10.0	10.0	10.0	10.0	10.
ACA	3.0	2.3	10.0	9.3	8.3	8.7	8.
	3.9	3.1	10.0	10.0	9.9	10.0	10.
	5.8	5.8	10.0	10.0	9.9	10.0	9.
	8.5	8.4	10.0	10.0	9.9	10.0	10.
	12.6	11.2	10.0	10.0	10.0	10.0	10.
NH ₄ OH-treated controls				4.0	0.8	0.0	0.0

Table 5. Results on red pine stakes installed at Ottawa, Ontario.

System Tested	Nominal Retention (kg/m ³)	Average Decay Ratings (Test time in years)			
		2	4	5	6
ACQ 1:1 (ODAC)	2.5	9.9	9.7	9.5	7.9
	4.6	9.9	9.9	9.9	9.4
	6.6	10.0	10.0	10.0	9.9
	8.5	10.0	10.0	10.0	9.9
	11.0	10.0	10.0	10.0	10.0
ACA	2.3	10.0	10.0	9.1	8.0
	3.9	10.0	10.0	9.9	9.5
	5.6	10.0	10.0	10.0	9.9
	7.2	10.0	10.0	10.0	9.7
	8.1	10.0	10.0	10.0	10.0
10.6	10.0	10.0	10.0	9.9	
Controls		7.2	1.4	0.0	0.0
NH ₄ OH-treated controls		4.0	0.4	0.0	0.0

Table 6. Southern yellow pine stake test data for Dorman, Miss. test site.

System Tested	Retentions		Average Ratings for Decay and Termites (Test time in years)					
			1		2		3	
	Nominal (kg/m ³)	Assay (kg/m ³)	Dec	Ter	Dec	Ter	Dec	Ter
ACQ 1:1 (ODAC)	3.3	3.4	10.0	10.0	9.9	9.9	9.1	9.1
	4.7	4.9	9.9	10.0	9.9	9.9	9.7	9.7
	6.9	6.2	10.0	10.0	9.9	10.0	9.8	10.0
	9.3	9.2	10.0	10.0	10.0	9.9	10.0	9.9
	14.8	13.2	9.9	10.0	9.9	10.0	9.9	10.0
ACQ 1:1 (DDAC)	3.5	3.3	10.0	10.0	10.0	10.0	8.8	9.1
	4.8	4.5	10.0	10.0	9.9	9.9	9.9	9.9
	6.7	6.5	10.0	10.0	9.9	10.0	9.8	10.0
	8.8	9.2	10.0	10.0	10.0	10.0	10.0	10.0
	13.0	12.0	10.0	10.0	10.0	10.0	9.9	10.0
CCA-C	2.6	2.7	10.0	10.0	10.0	10.0	10.0	10.0
	5.4	5.2	10.0	10.0	10.0	10.0	10.0	10.0
	8.1	8.0	10.0	10.0	10.0	9.9	10.0	9.9
Untreated controls			6.4	7.3	4.0	5.1	1.5	2.0

Table 7. Southern yellow pine stake test data for Saucier, Miss. test site.

		Average Ratings for Decay and Termites (Test time in years)							
		Retentions		1		2		3	
System Tested	Nominal (kg/m ³)	Assay (kg/m ³)	Dec	Ter	Dec	Ter	Dec	Ter	
ACQ 1:1 (ODAC)	3.1	3.3	10.0	10.0	9.5	9.9	9.0	9.1	
	4.7	4.8	10.0	9.9	9.4	9.9	9.0	9.5	
	6.8	6.1	10.0	10.0	10.0	9.9	9.9	9.9	
	9.4	9.3	10.0	10.0	10.0	9.9	10.0	9.9	
	14.5	14.9	10.0	10.0	10.0	10.0	10.0	10.0	
ACQ 1:1 (DDAC)	3.4	3.1	10.0	10.0	9.5	9.8	9.3	9.3	
	4.8	4.4	10.0	10.0	9.9	10.0	9.8	9.8	
	6.7	6.4	10.0	10.0	9.9	10.0	9.9	9.9	
	9.1	9.4	10.0	10.0	10.0	10.0	10.0	10.0	
	12.8	12.0	10.0	10.0	10.0	10.0	10.0	10.0	
CCA-C	2.6	2.7	10.0	10.0	10.0	9.9	10.0	9.7	
	5.4	5.2	10.0	10.0	10.0	9.9	10.0	9.9	
	8.1	8.0	10.0	10.0	10.0	9.9	10.0	9.9	
Untreated controls			3.1	5.2	0.3	1.5	0.0	0.0	

Table 8. Marine test results on red pine panels installed at Ponce Inlet, Florida

Ratings	Average Panel					
	Retentions		(Test time in years)			
	Nominal (kg/m ³)	Assay (kg/m ³)	1	3	4	5
ACQ (3:1) (ODAC)	9.3	7.5	10.0	9.6	7.0	3.4
	13.4	10.9	10.0	10.0	10.0	9.0
	18.9	13.8	10.0	10.0	10.0	9.2
	27.3	16.2	10.0	10.0	10.0	9.8
	35.9	26.5	10.0	10.0	10.0	9.8
ACQ (1:1) (ODAC)	12.3	11.2	10.0	9.6	7.8	6.2
	17.4	14.5	10.0	10.0	10.0	9.2
	25.6	19.3	10.0	10.0	10.0	10.0
	35.9	24.6	10.0	10.0	10.0	10.0
	45.9	33.7	10.0	10.0	10.0	10.0
ACQ (1:3) (ODAC)	16.5	11.1	10.0	10.0	4.2	2.6
	23.6	20.0	10.0	10.0	10.0	8.8
	32.1	25.8	10.0	10.0	9.8	8.8
	43.2	34.5	10.0	10.0	10.0	10.0
	61.8	37.1	10.0	10.0	10.0	10.0
CCA-C	12.5	14.5	10.0	9.8	9.8	9.6
	16.3	15.8	10.0	10.0	10.0	8.8
	25.6	25.0	10.0	10.0	10.0	10.0
	42.2	37.2	10.0	10.0	10.0	10.0
Marine creosote	132.2	138.1	9.2	4.2	2.2	1.4
	264.6	253.7	10.0	7.0	4.6	4.8
NH ₄ OH-treated controls			0.0	0.0	0.0	0.0

Table 9. Pole and timber treating results from plant trials.

Species	Commodity	M.C. (%)	Pressure period (hr)	Meeting Penet'n (%)	Assay Retention (kg/m ³)
ROUNDWOOD:					
Red pine	40 Class 4	65	9.8	92	14.5
Red pine	40 Class 4	27	6.0	96	12.2
Red pine	40 Class 4	75	6.0	83	12.6
Red pine	40 Class 4	27	1.0	68	9.3
Jack pine	40 Class 5	23	5.0	91	9.8
Jack pine	40 Class 5	51	5.0	93	9.4
Jack pine	40 Class 5	41	7.0	94	6.4
Jack pine	40 Class 5	26	12.3	94	6.0
Lodgepole	40 Class 4	19	9.0	80	9.4
Lodgepole	35 Class 5	24	7.0	66	8.6
Lodgepole	40 Class 3	19	7.0	86	10.7
Scots pine	40 Class 5	21	2.5	97	14.0
Scots pine	40 Class 5	19	4.0	98	9.2
TIMBER:					
E. Hemlock	8x8"x6'4"	94	5.0	78	8.0
E. Hemlock	8x8"x6'4"	94	2.0	90	11.2
E. Hemlock	8x8"x6'4"	89	4.0	98	9.9
Red pine	8x8"x6'4"	30	2.0	95	11.6