BONDABILITY OF SALVAGED YELLOW-CEDAR WITH PHENOL-RESORCINOL ADHESIVE AND HYDROXYMETHYLATED RESORCINOL COUPLING AGENT

E. ARNOLD OKKONEN
CHARES B. VICK †

ABSTRACT

A serious mortality problem affects about 65 percent of the estimated 2.02×10^6 m^2 (500×10^3 acres) of yellow-cedar in southeastern Alaska. These declining yellow-cedar stands contain a mixture of dead and living trees, and because of their great resistance to decay, these dead trees can remain standing for as long as 120 years after death. In the interest of better utilizing the affected timberland and the highly durable, potentially valuable yellow-cedar, a cooperative study was done to determine if deterioration had occurred in the mechanical properties of salvaged yellow-cedar. Faced with reports of unsatisfactory adhesive bonding from potential users and varying bondability reports of yellow-cedar from the United States Department of the Navy, Bureau of Ships, the study reported herein investigated whether phenol-resorcinol-formaldehyde (PRF) adhesive could develop durable bonds to the salvaged yellow-cedar. Test results of adhesive bonds in lumber laminates demonstrated that resistance to delamination, shear strength, and wood failure exceeded requirements of ASTM D 2559, which is a specification for adhesives used in structural laminated wood products exposed to exterior service conditions. Comparisons of data from this study and two studies by the Bureau of Ships indicate that bondability of yellow-cedar was not compromised because the lumber had been salvaged from 500-year-old trees that had been dead for 14 years. Wood products laminated from salvaged but durable yellow-cedar should command a premium in the marketplace, particularly for marine construction.

Yellow-cedar (*Chamaecyparis nootkatensis* (D. Don) Spach), also commonly known as Alaska-cedar, Alaska yellow-cedar, Nootka cypress, Sitka cypress, and yellow cypress, ranges along the Pacific Coast region, from south and southeast Alaska to west and southeast British Columbia and into the mountains of western Washington and Oregon (14). Yellow-cedar grows in cool and humid climates on the islands in British Columbia to Prince William Sound, Alaska, and in a narrow strip along the coastal mainland, usually no more than 241 km (150 miles) inland (8).

The estimated total volume of yellow-cedar sawtimber was 69.9×10^6 m^3 (29.6 $\times 10^9$ board feet) in the late 1970s. Much of this timber is inaccessible in high mountain ranges and poor sites, so even with the intense logging of today, reliable timber volumes are still not available. Long-term lumber production is not available either, because yellow-cedar is normally harvested in clear cuttings where it is mixed with other softwood species. Only small volumes of yellow-cedar are utilized domestically and in Canada; most of the harvest is exported to Japan (8).

A serious mortality problem affects an estimated 2.02×10^6 m^2 (500×10^3 acres) in southeast Alaska (13), where an average 65 percent of the basal area of yellow-cedar is dead (10). The primary cause for this mortality is not fully understood and cannot be ascribed to typical forest pathogens, insects, or nematodes (9,10,12). The species is very slow growing and long lived, so competing fast-growing species overcome yellow-cedar on the best-growing sites, relegating it to develop on thin organic soils over bedrock that is deficient in nutrients (8,10). Most declining yellow-cedar stands contain a mixture of living and dying trees and snags. (A snag is a standing dead tree, or a portion of a tree, from which most of the foliage and limbs have fallen.) These dead yellow-cedar trees vary from an average of 4 years since death (retained foliage) to more than 80 years since death (branches missing, top intact). The characteristic high level of decay resistance of the species retards deterioration of snags; therefore, some

The authors are, respectively, Physical Science Technician and Research Scientist, USDA Forest Serv., Forest Prod. Lab., One Gifford Pinchot Dr., Madison, WI 53705-2398. The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Dept. of Agriculture of any product or service. This paper was received for publication in October 1997. Reprint No. 8722.
† Forest Products Society Member.
© Forest Products Society 1998.
Forest Prod. J. 48(11/12):81-85.

remain standing for more than 120 years (10,11,15).

The yellow-cedar mortality problem was recognized, researched, and described by USDA Forest Service, Alaska Region, Pathologist Paul Hennon from the Pacific Northwest Research Station and Silviculturist John Stevens from the Tongass National Forest, Wrangell District. In their interest to better utilize the affected timberland and the highly durable, potentially valuable yellow-cedar, they sought assistance from the USDA Forest Service, Forest Products Laboratory (FPL). In a cooperative study with FPL Research Scientist Kent McDonald and Engineer Dave Green, the mechanical properties of salvaged yellow-cedar were measured and compared with properties of wood harvested from healthy trees (15). Initial property analyses showed no evidence that even the oldest yellow-cedar snags had lost strength. Therefore, McDonald and Green recommended that salvage and subsequent utilization of snags be based simply on the volume of sound logs and the normal expected yield of lumber. An additional study at FPL is underway to measure lumber recovery by volume and grade within each of the five snag classes, as well as to estimate recovery from other locations.

In their search for ways to utilize small cuttings and larger pieces of lumber, Alaska Forest Service scientists considered adhesive bonding a viable option. However, reports of unsatisfactory bonding had been received from potential users. In view of potential adhesion problems, our Forest Service cooperators asked us to investigate the bondability of the salvaged yellow-cedar. Our subsequent review of studies by the Bureau of Ships (5,6) revealed additional conflicting evidence of bonding problems with yellow-cedar.

BACKGROUND

In appearance and properties, the wood of yellow-cedar resembles Port-Orford-cedar and Atlantic white cedar. Yellow-cedar is strongly aromatic and has a high level of oil content. The heartwood is a distinctive, bright, sulfur yellow; the sapwood is lighter in color and difficult to distinguish from heartwood. The annual growth rings are barely visible because of very slow growth. The wood is finely textured and straight grained. Yellow-cedar has an average specific gravity of 0.42 and is moderate in strength, stiffness, hardness, and shock resistance (8).

Yellow-cedar is extremely durable. Extractives from the heartwood are solely responsible for the excellent durability of yellow-cedar (4). Among the chemical compounds extracted from the heartwood, Rennerfelt and Nacht (17) found that nootkatin, a tropolone, inhibited fungal growth at 0.001 to 0.002 percent concentration. Chamic acid, another heartwood extractive, inhibited fungi at 0.01 to 0.02 percent. The oily extractives tend to interfere with the absorption of oil-based paints and varnishes (4). Although not discussed in Barton's review (4), oily extractives may explain the reports of poor adhesion to the lumber. Primarily because of its great natural durability, as well as its resistance to acid, smooth-wearing quality, stability, and workability, yellow-cedar has a long history of use in construction of ships and smaller watercraft (8).

Given the great durability of yellow cedar under exterior service conditions, the United States Department of the Navy, Bureau of Ships, investigated the bondability of the wood with highly durable phenol-resorcinol-formaldehyde (PRF) adhesives (5,6). The Navy wanted bonds in yellow-cedar lumber laminates that would equal the durability of the wood. Durable, laminated wood products that could be fabricated from smaller pieces of wood and shaped to almost any configuration would be particularly useful in marine construction and command a premium in the marketplace.

Conclusions from the Navy's two studies (5,6) on the bondability of yellow-cedar with PRF adhesives were surprisingly varied, particularly in view of the similarity of adhesives, specimen preparation, and testing procedures. Adhesive curing procedures in both studies were conducted at elevated temperatures according to United States Department of the Navy specifications. The Timber Engineering Company, under contract from the Bureau of Ships (6), reported that PRF adhesives developed only moderately good bonds, as determined from block-shear strength and wood failure. Half the laminated beams had shear strength values less than the allowable. One adhesive produced moderately good resistance to delamination, and the other produced high resistance. After 1 year of outdoor exposure, beam sections performed satisfactorily. Apparently dissatisfied with the outcome of this study, the Bureau of Ships (5) conducted a study using materials, specimen preparation, and testing procedures similar to that of the Timber Engineering Company's study. Results of the Bureau of Ships investigation indicated that shear strength, wood failure, and resistance to delamination were well above minimum requirements for adhesives covered by Specification MIL-A-397B (5).

OBJECTIVES

The primary objective of the study reported herein was to determine if a commercial PRF adhesive could develop bonds to salvaged yellow-cedar lumber that would meet the requirements for shear strength, wood failure, and resistance to delamination, specified in ASTM D 2559 (3). This specification is used to qualify adhesives for structural glued-laminated timbers intended for wet-use exposures under industry standard ANSI/AITC A190.1-1992 (1). A secondary objective was to determine if a recently discovered hydroxymethylated resorcinol (HMR) coupling agent (18, 19) would enhance adhesion of the PRF adhesive to yellow-cedar, should there be adhesive-wood incompatibility as prior studies indicated. HMR has been shown to substantially improve adhesion of several thermosetting wood adhesives, including PRF, to several species of wood, including preservative-treated wood, when tested according to ASTM D 2559 (3).

MATERIALS

WOOD

Lumber for specimens was sawn from two, 122-cm- (48-in.-) long bolts that were cut from sample snags, 48 to 58 cm (18.9 to 22.8 in.) in diameter. Bolts came from snag class II, meaning that the standing trees had been dead an average of 14 years but had retained twigs with missing dead foliage. The trees from which the bolts were cut were 480 to 500 years old, with growth rates ranging from 16 to 21 rings/cm (41 to 53 rings/in.). Snags were collected by McDonald (FPL) and Hennon (Alaska Region) in 1995 from a site near Nemo Point on the west side of Wrangell Island in the Stikene Area of Tongass National Forest in southeast Alaska (15).

The lumber was quartersawn (radial grain) because the material had been selected and cut for testing mechanical

properties according to the requirements of the ASTM test method for small, clear specimens. Flatsawn lumber is required for delamination and block-shear tests in ASTM D 2559 (3). Therefore, shear strength, wood failure, and delamination of bonds to the quarter sawn lumber may differ somewhat from what they might have been on flatsawn lumber. However, such grain effects probably would be minimized by the extremely slow growth that makes the annual rings barely visible.

All lumber was conditioned to approximately 9.5 percent equilibrium moisture content (EMC) at 22.8°C (73°F) and 50 percent relative humidity.

ADHESIVE

A two-component PRF adhesive with Penacolite G-4422 resin and Penacolite G-4400-B hardener, manufactured by Indspec Chemical Corporation of Pittsburgh, Pa., was used for all bonding tests. The resin and hardener were mixed in a 5:1 ratio by weight. This adhesive is certified by its manufacturer as meeting the requirements of ASTM D 2559 (3). The adhesive cures at room temperature, has a pot life from 2 to 3 hours, and requires pressure for 6 to 9 hours during cure. The manufacturer's instructions were followed in proportioning, mixing, and applying the adhesive.

HMR COUPLING AGENT

A 5 percent solid aqueous solution of HMR coupling agent was used to prime yellow-cedar surfaces before bonding with PRF adhesive (18,19). The ingredients listed in **Table 1** were reacted at room temperature for 4 hours before applying to the wood surfaces. The length of this reaction time is critical to the effectiveness of adhesion. Reaction time should not be less than 4 hours nor more than 6 hours. Dodecyl sulfate sodium salt (0.5% by weight) was added to the HMR primer solution at the end of the 4-hour reaction time to aid wetting of yellow-cedar.

TABLE 1.–Ingredients for HMR coupling agent.

Ingredients	Parts by weight
Water, deionized	90.43
Resorcinol, crystalline	3.34
Formaldehyde, 37 percent formalin	3.79
Sodium hydroxide, 3 molar	2.44
Total	100.00

EXPERIMENTAL PROCEDURES

STATISTICAL ANALYSIS

This experiment was designed to determine shear strength, wood failure, and delamination of a PRF adhesive in laminated lumber joints of unprimed yellow-cedar and to compare these results with similarly prepared joints with lumber surfaces primed with an HMR coupling agent. A t-test for unpaired replicates was used to compare the three properties from unprimed and primed lumber joints (7). For shear strength and wood failure tests, 20 block-size specimens were prepared from four, two-ply lumber laminates, representing each of the unprimed and primed groups. For delamination tests, six delamination specimens were prepared from two, six-ply lumber laminates, representing each of the two groups. Delamination was measured from five bondlines on each end of the six delamination specimens for a total bondline length of 419 cm (165 in.) for each group.

SPECIMEN PREPARATION

Shear strength and wood failure were determined by compression-loaded, block-shear specimens with a 19.4-cm^2 (3.0-$in.^2$) shear area. The specimens were prepared from two-ply lumber laminates and cut into block-shear specimens as described in ASTM D 905 (2). Each lumber laminate measured 1.9 cm (3/4 in.) thick, 6.4 cm (2-1/2 in.) wide, and 30.5 cm (12 in.) long.

Resistance to delamination was determined from cross sections, 7.6 cm (3 in.) long, cut from six-ply lumber laminates. A laminate was prepared by bonding together six pieces of lumber, each measuring 1.9 cm (3/4 in.) thick, 7.6 cm (3 in.) wide, and 31.5 cm (12 in.) long.

Two- and six-ply lumber laminates were prepared in the same manner. If lumber surfaces were to be primed before bonding, a 5 percent HMR solution was spread on both surfaces with a brush at approximately 0.15 kg/m^2 (0.03 psf). The primed surfaces were dried 24 hours at 22.8°C (73°F) and 50 percent RH. The adhesive was spread with a roller on both bonding surfaces to total 0.39 kg/m^2 (0.08 psf). Closed assembly time was approximately 15 minutes, then pressure of 690 kPa (100 psi) was applied for 24 hours at room temperature.

TESTING PROCEDURES

Block-shear specimens were tested for dry shear strength and wood failure according to ASTM D 905 (2). Shear strength at failure was calculated based on a shear area of 19.4 cm^2 (3.0 $in.^2$). Wood failure in the shear area was estimated to the nearest 5 percent.

Delamination specimens were subjected to the following three cycles of the delamination test in ASTM D 2559 (3):

Cycle 1

1. Vacuum soak in water at 84.4 kPa (25 in.-Hg) for 5 minutes.
2. Pressure soak in water at 517 kPa (75 psi) for 1 hour.
3. Repeat events 1 and 2.
4. Dry at 65.5°C (150°F) for 21 to 22 hours.

Cycle 2

1. Steam at 100°C (212°F) for 1-1/2 hours.
2. Pressure soak in water at 517 kPa (75 psi) for 40 minutes.
3. Repeat event 4 from Cycle 1.

Cycle 3

1. Repeat events in Cycle 1.

Immediately after the final cycle, delamination was measured along the end-grain surfaces to the nearest 0.25 mm (0.01 in.) with a machinist's scale under a stereomicroscope. Delamination was ex-

TABLE 2.–Resistance to delamination of PRF adhesive bonds in HMR-primed and unprimed salvaged yellow-cedar laminates.

Surface primer	Resistance to delamination				
	Mean	Minimum	Maximum	Coefficient of variation	Statistical comparison
	(%)				
None	3.6	0	7.3	0.89	A[a]
HMR	1.4	0	2.8	0.75	A
ASTM D 2559[b]	≤ 5.0	--	--	--	--

[a] Comparison of means for HMR-primed and unprimed yellow-cedar laminates; means with the same capital letter are not significantly different at the 0.05 level of probability.
[b] Requirement of ASTM D 2559.

TABLE 3.–Shear strength and wood failure for HMR-primed and unprimed salvaged yellow-cedar laminates bonded with a PRF adhesive.

Surface primer	Shear strength					Wood failure				
	Mean	Minimum	Maximum	Coefficient of variation	Statistical comparison[a]	Mean	Minimum	Maximum	Coefficient of variation	Statistical comparison[a]
	(N/cm^2 (psi))					(%)				
None	1142	971	1298							
	(1,656)	(1,408)	(1,883)	0.069	A	95	75	100	0.060	A
HMR	1096	963	1254							
	(1,590)	(1,397)	(1,819)	0.077	A	90	80	100	0.085	A
ASTM D 2559[b]	≥ 826									
	(1,198)	—	--	--	--	≥ 75	--	--	--	--

[a] Comparison of means for HMR-primed and unprimed yellow-cedar laminates; means with the same capital letter are not significantly different at the 0.05 level of probability.
[b] Minimum requirements of ASTM D 2559.

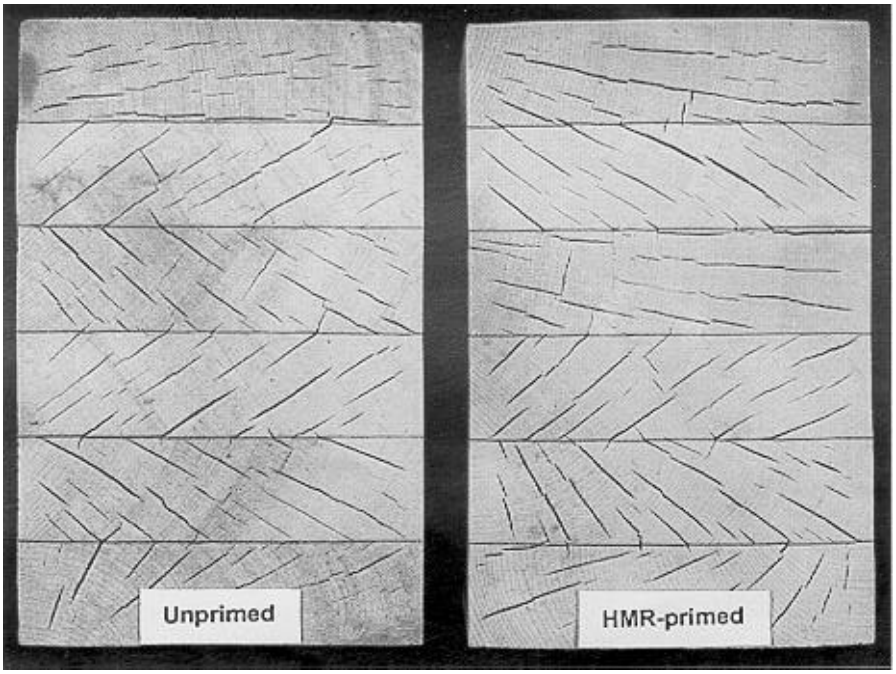

Figure 1. – Severe checking but no delamination on the ends of unprimed and HMR-primed yellow-cedar laminates after the cyclic delamination test of ASTM D 2559 (3).

pressed as a percentage of total end-grain bondline length for each specimen.

RESULTS AND DISCUSSION

RESISTANCE TO DELAMINATION

Resistance to delamination, as specified in ASTM D 2559 (3) is the most rigorous of tests used to determine durability of adhesives in structural laminated wood products intended for exterior service. Delamination of the room-temperature-cured PRF adhesive bonds in unprimed and HMR-primed, salvaged yellow-cedar lumber laminates, is shown in **Table 2.** Data show that whether or not lumber is primed with HMR, the PRF adhesive met the 5 percent maximum delamination requirement of ASTM D 2559 (3). Average delamination on the unprimed lumber was 3.6 and 1.4 percent on the HMR-primed lumber. A statistical t-test showed no significant differences in average delamination percentages between the two surface treatments. End-grain surfaces of unprimed and HMR-primed delamination specimens after the accelerated three-cycle delamination test are shown in **Figure 1.** Note the severe checking within the wood and absence of delamination in the bondlines of both specimens.

Delamination data from this experiment can be compared with that of Finnom (6). However, note that adhesives, specimen preparation, testing procedures, and curing temperature in Finnom's study were quite different from the materials and methods used in our study. Finnorn prepared and tested specimens according to Specification MIL-W-2038 B (Ships), which requires curing adhesive at 65.5°C (150°F) for 6 hours and subjects delamination specimens to a three-cycle, 12-day water-soak and drying test. Curing at elevated temperature ensures deep adhesive penetration and complete chemical cure. The 12-day Navy delamination test is less severe than the 3-day ASTM delamination test. Under these conditions, Finnorn's Penacolite G- 1260 PRF adhesive delaminated only 0.2 percent (6). Using the same adhesive and similar specimen preparation and testing procedures, England et al. (5) reported 0.7 percent delamination.

SHEAR STRENGTH AND WOOD FAILURE

The shear strength of unprimed block-shear specimens averaged 1142 N/cm^2 (1,656 psi), with a range from 971 N/cm^2 (1,408 psi) to 1298 N/cm^2 (1,883 psi) **(Table 3).** The strength values of HMR-primed bonds averaged 1096 N/cm^2 (1,590 psi), ranging from 963 N/cm^2 (1,397 psi) to 1254 N/cm^2 (1,819 psi). Average shear strength values of unprimed and HMR-primed joints were not significantly different. The average strength values of both groups, as well as the lowest values, exceeded the minimum 826 N/cm^2 (1,198 psi) required by ASTM D 2559 (3).

Shear strength for salvaged yellow-cedar in this experiment (1142 N/cm^2 (1,656 psi) **(Table 3)** was 76 percent greater than that reported by Finnorn (6) (649 N/cm^2 (941 psi)) and 11 percent

greater than that reported by England et al. (5) (1033 N/cm^2 (1,498 psi)). The fact that shear strength values by Finnorn were below standard requirements probably occasioned the subsequent study by England et al. Again, the differences between materials and methods of preparing and testing of specimens should be considered. In particular, the grain orientation was radial to the face of the lumber in our study, but was tangential in both of the cited studies. It is generally true that shear strength of bonded radial-grained softwood lumber is considerably greater than flat-grained lumber (16).

Wood failures from unprimed and HMR-primed block-shear specimens were 95 and 90 percent, respectively **(Table 3).** The differences between these averages were not statistically significant. These averages were 27 and 20 percent greater, respectively, compared to the minimum 7.5 percent required by ASTM D 2559 (3). Wood failure of the unprimed, salvaged yellow-cedar (95%) was 13 percent greater than the 84 percent reported by Finnom (6) and approximately the same as that determined by England et al. (5).

Conclusions

The bondability of salvaged yellow-cedar with a room-temperature cured, PRF adhesive was excellent. Resistance to delamination, shear strength, and wood failure exceeded the requirements of ASTM D 2559, which is a specification for adhesives used in structural laminated wood products exposed to exterior service conditions. It was not necessary to prime lumber surfaces of the salvaged wood with an HMR coupling agent to meet these adhesion requirements. Should other yellow-cedar lumber prove less amenable to adhesion by PRF adhesives, then HMR is a viable means of improving bondability. Comparisons of data from salvaged yellow-cedar in our study with that from lumber from live trees in past studies sponsored by the United States Department of Navy, Bureau of Ships, indicated that bondability of yellow-cedar was not compromised because the lumber had been salvaged from 500-year-old trees that had been dead 14 years.

Literature Cited

1. American Institute of Timber Construction. 1992. American national standard for wood products: Structural glued laminated timber. ANSI/AITC A190.1-1992. AITC, Englewood, Colo. 16 pp.
2. American Society for Testing and Materials. 1996. Standard test method for strength properties of adhesive bonds in shear by compression loading. ASTM D 905-94. Annual Book of ASTM Standards, 15.06. ASTM, West Conshohocken, Pa. pp. 21-24.
3. _____. 1996. Standard specification for adhesives for structural laminated wood products for use under exterior (wet-use) exposure conditions. ASTM D 2559-92. Annual Book of ASTM Standards, 15.06. ASTM, West Conshohocken, Pa. pp. 154-158.
4. Barton, G.M. 1976. A review of yellow-cedar (*Chamaecyparis nootkatensis* (D. Don) Spach) extractives and their importance to utilization. Wood and Fiber 8(3):172-176.
5. England, R.F., E. Stahl, and W.H. Miles. 1961. Marine laminating properties of selected wood species: Alaska yellow-cedar. Prog. Rept., Dept. of the Navy, Bureau of Ships, E-412-15. Materials Lab., Puget Sound Naval Shipyard, Bremerton, Wash. 11 pp.
6. Finnorn, W.J. 1957. Gluability of Alaska yellow-cedar. Contract NObs 72024. Index No. NS-032-001. Dept. of the Navy, Bureau of Ships, Bremerton, Wash. 22 pp.
7. Freas, F. 1959. A guidebook for statistical transients. USDA Forest Serv., Southern Forest Expt. Sta. pp. 13-14.
8. Harris, A.S. 1984. Alaska-cedar: An American wood. FS-224. USDA Forest Serv., Washington, D.C. 6 pp.
9. Hennon, P.E. 1990. Fungi on *Chamaecyparis nootkatensis*. Mycologia 82:59-66.
10. _____, E.M. Hansen, and C.G. Shaw III. 1990. Dynamics of decline and mortality of *Chamaecyparis nootkatensis* in southeast Alaska. Canadian J. of Botany 68:651-662.
11. _____, C.G. Shaw III, and E.M. Hansen. 1990. Dating decline and mortality of *Chamaecyparis nootkatensis* in southeast Alaska. Forest Sci. 36:502-515.
12. _____, _____, and _____. 1990. Symptoms and fungal associations of declining *Chamaecyparis nootkatensis* in southeast Alaska. Plant Disease 74:267-273.
13. Holsten, E., P.E. Hennon, D. Witter, and K. Mathews. 1996. Forest insect and disease conditions in Alaska, 1996. R10-TP-61. USDA Forest Serv., State and Private Forestry, Forest Health Management, Anchorage, Alaska. 40 pp.
14. Little, E.L., Jr. 1979. Checklist of United States trees (native and naturalized). Handbook 541. USDA Forest Serv., Washington, D.C. 87 pp.
15. McDonald, K.A., P.E. Hennon, J.H. Stevens, and D.W. Green. 1997. Mechanical properties of salvaged dead yellow-cedar in southeast Alaska: Phase I. FPL-RP-565. USDA Forest Serv., Forest Prod. Lab., Madison, Wis.
16. Okkonen, E.A and B.H. River. 1989. Factors affecting the strength of block-shear specimens. Forest Prod. J. 39(1):43-50.
17. Rennerfelt, E. and G. Nacht. 1955. The fungicidal activity of some constituents from heartwood of conifers. Sven. Bot. Tidskr. 49(3):419-432.
18. Vick, C.B. 1995. Coupling agent improves durability of PRF bonds to CCA-treated southern pine. Forest Prod. J. 45(3):78-84.
19. _____, K. Richter, and B.H. River. 1996. Hydroxymethylated resorcinol coupling agent and method for bonding wood. U.S. Patent No. 5,543,487. August 6. U.S. Dept. of Agriculture, assignee.