EVALUATION OF CHLOROSULFONYL PYRIDINE FOR PROTECTING WOOD FROM SOFT-ROT FUNGI USING A TENSILE STRENGTH TEST[1]

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ABSTRACT

Laboratory evaluation of a promising new wood preservative, 2,3,5,6,-tetrachloro-4 methylsulfonyl pyridine, was conducted to determine its relative effectiveness against two soft-rot fungi using tensile strength as the measure of the protection value. One-sixteenth-inch sapwood specimens of three species: ponderosa pine (*Pinus ponderosa* Laws.), sweetgum (*Liquidambar styraciflua* L.), and pondcypress (*Taxodium ascendens* Bong.); were treated with various levels of preservative and exposed to *Graphium* sp. and *Acremonium* sp. in test tube incubation chambers for 14 days. Statistical comparison of strength values of treated sweetgum specimens exposed to *Graphium* sp. indicated no significant difference between the protective properties of chlorosulfonyl pyridine and pentachlorophenol. Multiple comparisons of the untreated unincubated specimen mean with treated specimen means, however, indicated that 0.05 pcf of chlorosulfonyl pyridine was required to provide the same protection as afforded by 0.03 pcf of pentachlorophenol. Results also revealed that *Graphium* sp. had a relatively high wood-deterioration capacity.

Keywords: *Pinus ponderosa*, *Liquidambar styraciflua*, *Taxodium ascendens*, wood preservation, wood preservatives, retention, soft-rot, *Acremonium*, *Graphium*, pentachlorophenol, biodegradation, tensile strength.

INTRODUCTION

An essential step in developing a new wood preservative is laboratory evaluation of decay or deterioration occurring in wood specimens treated to contain various amounts of the test chemical. Treated wood specimens are inoculated with pure cultures of decay fungi and the resulting amounts of decay are evaluated by weight loss, strength reduction, or other methods (Behr 1973). Loss in strength due to decay often occurs before significant weight loss; therefore, any procedure that uses strength loss as an indicator of wood decay involves less incubation time, 12 weeks being required when using the ASTM soil-block method versus several weeks when strength loss is used. For this reason, strength loss has frequently been proposed for use in laboratory evaluation of wood decay (Brown 1963; Hartley 1958; Kennedy 1958; Mulholland 1954; Toole 1971).

Soft-rot fungi characteristically deteriorate the surface layers of susceptible wood when the latter is exposed in extremely wet and otherwise favorable situations. The manner in which the soft-rot fungi attack wood is different from that of common wood decay Basidiomycetes. Soft-rot fungi have a number of physiological and ecological characteristics that are distinctive. The importance and uniqueness of their attack in wood were not recognized until 1950 and later (Findlay and Savory 1950; Savory 1954).

This paper reports the effectiveness of a promising new wood preservative, 2,3,5,6-tetrachloro-4 methylsulfonyl pyridine,[3] rela-

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[3] Dowicil S-13, U.S.D.A. Registration No. 404-353, is a registered trademark of The Dow Chemical Company.

tive to that of pentachlorophenol[4] for protecting wood against biodeterioration caused by the soft-rot fungi, *Graphium* sp. and *Acremonium* sp. on sapwood specimens of ponderosa pine (*Pinus ponderosa* Laws.), sweetgum (*Liquidambar styraciflua* L.), and pondcypress (*Taxodium ascendens* Bong.). Tensile strength parallel to the grain was the criterion for the laboratory evaluation of deterioration.

MATERIALS AND METHODS

Test specimens were prepared from flat-sawn sapwood planks of the three wood species. These planks were freshly sawn and kiln-dried. The character and dimensions of the specimens are shown in Fig. 1; dimensions were identical to those used by Brown (1963). Specimens tested included only those that were free from defect, even-textured, straight-grained, and of accurate dimensions. The wood-preserving chemicals were prepared in concentrations needed to produce the following retentions: 0.01, 0.03, 0.05, and 0.07 pounds per cubic foot (pcf) of wood upon completion of treatment. The fungal isolates[5] used were a) *Graphium* sp., Designation: MCX-11 (R-47F); b) *Acremonium* sp., Designation: MDX-1 (R-11). According to Duncan (1960) both of these soft-rot isolates showed substantial wood-deteriorating capacities under a variety of test conditions. The culture medium was agar substrate which contained the ingredients recommended by Duncan (1965).

Two similar but separate experiments were conducted, one for each of the test fungi. Each experiment followed a factorial randomized block design to compare two wood preservatives at four retention levels using three wood species. Three replicates of each condition (preservative treatment-wood species combination) were included. For each wood species, there were nine untreated controls. To minimize strength variation within the test, observations of a given wood species were assigned to three blocks so the specimens within each block originated from the same annual rings and the same longitudinal position or section (Fig. 1A) in the plank. Each of the three blocks was comprised of one specimen to serve as the untreated incubated control, eight specimens for the preservative treatments, and three specimens (untreated and unincubated) for use to estimate the normal (undecayed) tensile strength for the group. Thus each block contained twelve observations. Within each block, wood specimens were assigned randomly to various treatments.

Wood specimens were conditioned at 70% RH and 80 F temperature in humidity jars similar to those described by Lin (1971). After removal from the humidity jars, the specimens were measured to obtain minimum cross-sectional areas. Subsequent preservative treatment and weathering of wood specimens were accomplished in accordance with A.S.T.M.-D-1413 (1970) with minor modifications, including the use of a larger beaker to accommodate the specimen and conditioned air instead of a water bath to maintain the required temperature. After weathering, the specimens were autoclaved at 140 F under atmospheric pressure for six hours.

Incubation chambers (Fig. 2) were prepared similar to those used by Brown (1963) except for a modification to favor soft-rot attack. This modification involved the addition of 15 ml of sterilized water to each chamber to increase the moisture content of the wood specimens. According to Duncan (1960, 1965) wood having a high moisture content is necessary for soft-rotting. To contain the water at a proper level, with the ends of the test specimens partially submerged, the incubation chambers were placed on incubator shelves inclined 10 degrees from the horizontal. A preliminary test indicated that the optimum temperature for growth of both fungi was between 34 and 36 C. The test specimens were placed in contact with the growing

[4] Dowicide 7, Prilled Technical Grade, U.S.D.A. Registration No. 464-72, is a registered trademark of The Dow Chemical Company.

[5] Obtained through courtesy of the U.S. Forest Products Laboratory, Madison, Wisconsin.

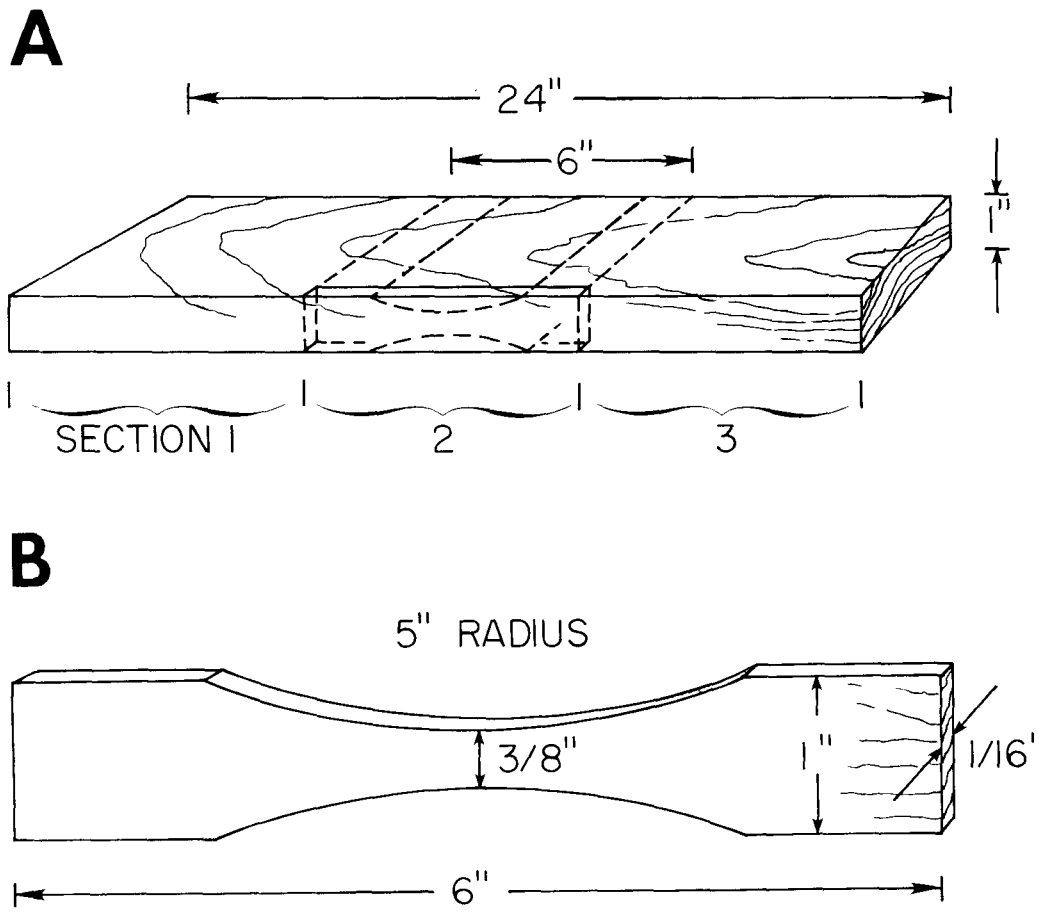

FIG. 1. Sapwood tensile test specimens. A. Flat-sawn plank with dotted lines indicating machining required to produce test specimens. Specimens from longitudinal sections 1, 2, and 3 were assigned to corresponding statistical blocks. B. Dimensions of specimens.

culture and subjected to fungal attack for 14 days at a temperature of 34 C.

Upon removal from the incubation chambers, the surfaces of the specimens were brushed clean. The specimens were then conditioned in the same manner as during initial conditioning. Subsequently the specimens were loaded in tension parallel-to-the-grain using a static timber-testing machine. The testing room was maintained at 70% RH and 80 F temperature. Rate of loading is a critical variable when testing the strength of wood (King 1961; Markwardt and Liska 1956; Sugiyama 1967). It was desirable, as shown by Brown (1963), to use a deformation rate resulting in maximum breaking load, thereby providing a maximum range of tensile strength from soft-rotted to sound specimens. A preliminary test determined the optimum loading speed to be 0.1 inch per minute. The ultimate load, in pounds, and the nature of the failure were recorded for each specimen. Load data were converted to tensile strength (psi) and plotted against retention. Comparison of preservative treatments was based upon statistical analysis.

RESULTS AND DISCUSSION

The average tensile strength values of the tested specimens are presented in Table 1. Among all treatments within each wood

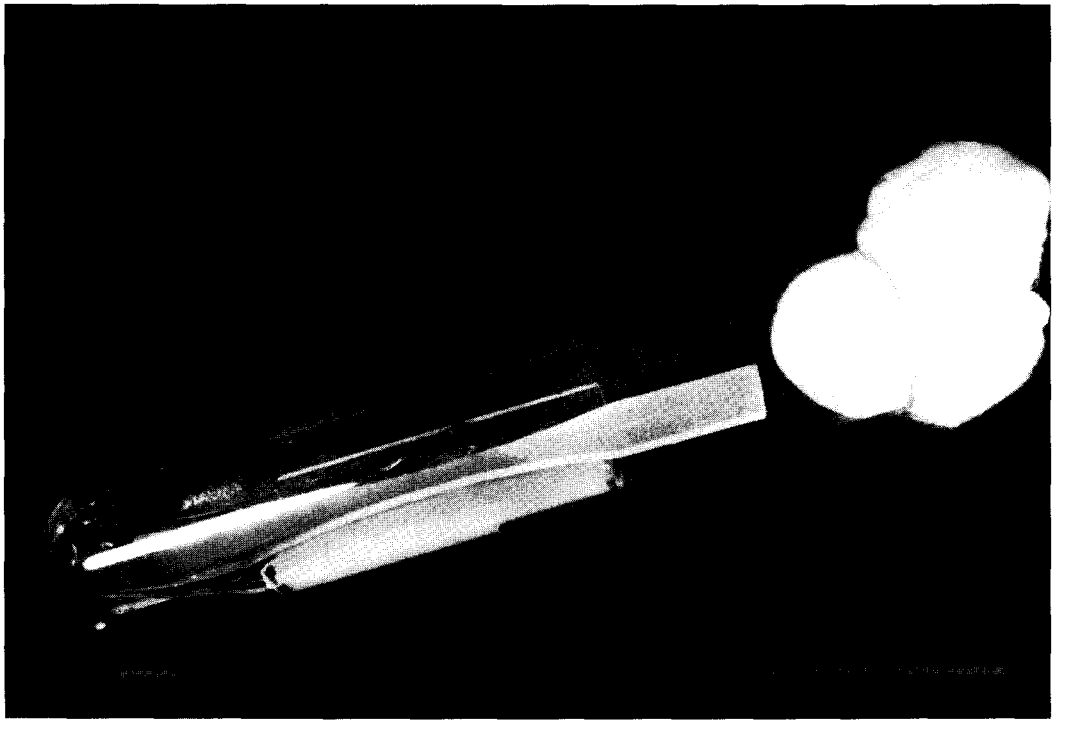

FIG. 2. Incubation chamber containing water, a soft-rot fungus, and a sapwood specimen. The chamber is inclined 10 degrees with the lower portion of the wood specimen submerged. The wood is in contact with the fungus growing on agar contained in a glass insert.

species-fungus category, highest tensile strengths were exhibited by the untreated unincubated controls. Coefficients of variations among these controls in the *Graphium* sp. experiment for ponderosa pine, sweetgum, and pondcypress were 4.8, 10.4, and 14.9 percent, respectively. These coefficients provide an indication of the original (normal) variation among the different species of test wood specimens. Lowest tensile strengths, with several exceptions, occurred among the untreated incubated (soft-rotted) controls.

The relative degree of deterioration of the various treated specimens caused by the test fungi is indicated by loss of strength of these specimens when compared to the strengths of the untreated unincubated controls. Such comparisons (Table 1) indicate that significant deterioration was caused by *Graphium* sp., with greatest strength loss occurring among sweetgum and ponderosa pine species. *Acremonium* sp. under conditions of this experiment caused limited strength reduction among the treated and untreated test specimens of all three test woods, a somewhat unexpected result in view of the earlier findings of Duncan (1960).

Further insight regarding the relative deteriorating effects caused by the test fungi and information on their deterioration capacities were gained by calculating and comparing average strength reduction data of the untreated specimens. Such strength reductions calculated from differences in tensile strengths of unincubated and incubated controls (Table 1) are presented in Table 2. These data show that among all wood species-fungus groups, *Graphium* sp. attack on sweetgum sapwood caused the greatest reduction in tensile strength; specifically, 10.08 psi $\times$ 10^{-3}, or 57.9%. The attack of the same fungus on ponderosa

TABLE 1. *Average tensile strength (psi × 10^{-3}) of sapwood specimens[a] exposed to various preservative and soft-rot fungus treatments.*

Wood Species	Untreated Incubated Controls[b]	Treated Incubated Specimens[b]								Untreated Unincubated Controls[c]
		Chlorosulfonyl Pyridine Retentions (pcf)				Pentachlorophenol Retentions (pcf)				
		0.01	0.03	0.05	0.07	0.01	0.03	0.05	0.07	
	----------Exposed to *Graphium sp.* for 14 days----------									
Ponderosa pine	7.62	9.96	9.55	9.60	9.21	9.31	9.60	9.36	9.86	10.15
Sweetgum	7.33	7.82	11.28	14.37	15.74	9.36	15.77	15.77	15.41	17.41
Pondcypress	11.76	10.84	11.10	11.37	11.84	12.20	13.00	12.50	11.93	12.67
	----------Exposed to *Acremonium sp.* for 14 days----------									
Ponderosa pine	7.36	7.84	8.21	7.64	8.33	8.22	8.36	7.44	7.33	7.70
Sweetgum	15.06	14.79	14.36	16.83	15.32	15.88	14.73	14.35	14.76	16.67
Pondcypress	12.78	14.23	12.86	12.71	12.26	13.00	12.99	13.70	12.31	12.98

[a]Specimens were weathered and reconditioned at 70% RH and 80°F temperature prior to tensile testing; retentions are those at time of treatment.

[b]Mean of 3 determinations.

[c]Mean of 9 determinations.

pine resulted in a 24.9% strength reduction. On the basis of past experience, strength reductions of about 60% or higher are usually observed among untreated controls which exhibit significant levels of Basidiomycete decay (Brown 1963).

The apparent limited effects of *Acremonium* sp. on untreated controls led to the conclusion that comparisons of the two test chemicals could be based only on the data from sweetgum exposed to *Graphium* sp. This conclusion was confirmed when results of the analysis of variance of the treatments among all other wood species-fungus categories were determined to be non-significant. The analysis of variance of tensile strength values of sweetgum species exposed to *Graphium* sp. is presented in Table 3. An "F" value of 4.77 indicated significant differences at the 0.01 level among treatments, and thus the data provided a valid basis for testing the effectiveness of the test chemical.

Subsequent statistical analysis revealed no significant difference between the protective properties of chlorosulfonyl pyridine and pentachlorophenol when they were used to protect wood against soft-rot fungus. In such analysis the calculated "t" value obtained from the comparison of ten-

TABLE 2. *Average strength reduction[a] and percent reduction[b] of untreated sapwood specimens exposed to soft-rot fungi.*

Wood Species	Soft-Rot Fungi			
	Graphium sp.		*Acremonium sp.*	
	psi x 10^{-3}	%	psi x 10^{-3}	%
Ponderosa pine	2.53[a]	24.9[b]	0.34	4.4
Sweetgum	10.08	57.9	1.61	9.7
Pondcypress	0.91	7.2	0.20	1.5

[a]Tensile strength differential between unincubated and incubated controls.

[b]Tensile strength reduction expressed as percentage of unincubated control tensile strength.

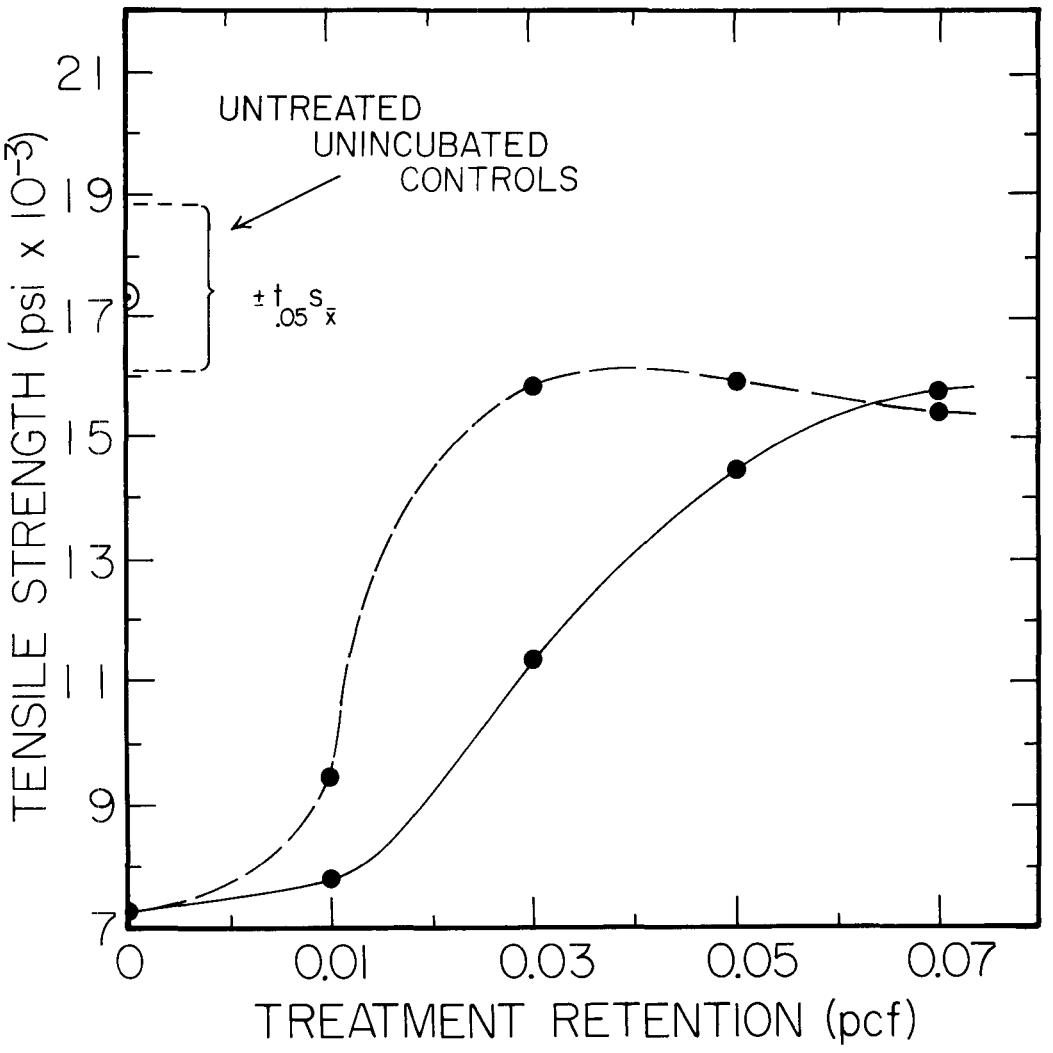

FIG. 3. Average tensile strength of weathered sweetgum specimens as affected by various retention levels of two preservative chemicals, pentachlorophenol (- - - -) and chlorosulfonyl pyridine (——), and 14-day exposure to *Graphium* sp. compared with tensile strength of the unincubated controls.

sile strength values of chlorosulfonyl pyridine versus pentachlorophenol on sweetgum specimens in the *Graphium* experiment was 1.47, the table "t" value indicating a significant difference for the comparison was 2.10.

Employing Dunnett's multiple comparison procedure (Steele and Torrie 1960) to compare the untreated, uninoculated sweetgum control mean (17.41×10^{-3}) with each of the eight treated incubated (*Graphium* sp.) means provides a statistical method to determine for each preservative the lowest effective retention level needed to protect wood. For example, the four means representing the tensile strength of specimens treated with chlorosulfonyl pyridine at the four different retention levels were

7.82, 11.28, 14.37, and 15.74 psi $\times 10^{-3}$, respectively.

Only two of these, 7.82 and 11.28, were

TABLE 3. *Analysis of variance of tensile strengths of sweetgum specimens exposed to Graphium sp.*

Source of Variation	d.f.	S.S.	M.S.	F
Blocks	2	56.42×10^6		
Treatments	9	375.57×10^6	41.73×10^6	4.77**
Error	18	157.40×10^6	8.74×10^6	
Total	29	589.39×10^6		

**Significant at 0.01 level.

found to be significantly different from 17.41; the other two, 14.37 and 15.74, were not significantly different. Since 14.37 represented the strength of specimens treated with 0.05 pcf of chemical, this retention level for chlorosulfonyl pyridine under conditions of this study is the lowest effective retention level that will prevent degradation of sweetgum sapwood by *Graphium* sp. Using the same procedure, the lowest effective retention level for pentachlorophenol to protect sweetgum sapwood was 0.03 pcf.

Tensile strength data of the chlorosulfonyl pyridine and pentachlorophenol specimens plotted against retention at time of treatment are presented in Fig. 3. Mean tensile strength and confidence limits ($\pm t_{.05}s_{\bar{x}}$) of the unincubated treated controls are shown for comparison. Data for the treated, weathered, and incubated specimens in Fig. 3 suggested that at the midpoint of the tensile strength range, chlorosulfonyl pyridine as a preservative for protecting wood against *Graphium* sp. attack was approximately three-fourths as effective as pentachlorophenol when based on retention at time of treatment.

As previously indicated, strength reductions caused by *Acremonium* sp. were limited. The attack of this isolate on the untreated incubated control specimens was insufficient to provide a basis for evaluating the preservative value of chlorosulfonyl pyridine. The test results provide additional information, however, on the wood-deterioration capacity of the fungus, at least under conditions used in this experiment. Extending the incubation period, decreasing the wood moisture content or other changes in technique will undoubtedly be necessary to enable this isolate to cause significant damage when similar studies are conducted.

Tensile strength testing is sensitive in evaluating limited deterioration and is recommended when soft-rot fungi are employed. The tensile strength method has the disadvantage of being a destructive test. As such, it is impossible to measure strength loss directly because the normal tensile strength of each soft-rotted specimen is an estimated value based on the average strength value of unincubated controls. Since an additional variable—strength of wood—is added to the test, factors that influence the strength properties of wood must be critically controlled.

In order to obtain satisfactory information for fungistatic evaluation, a fungus isolate having a high deterioration potential should be selected for testing wood preservatives. Woods selected should be of uniform texture and highly susceptible to soft-rotting. The combination of *Graphium* sp. and sweetgum sapwood appear to have these attributes and therefore may be useful for evaluating wood preservation when the tensile test method is employed. Pond-cypress sapwood, on the other hand, is not a satisfactory material for tensile testing because of its relatively uneven texture and low susceptibility to deterioration by this isolate.

The patterns of wood failure in the specimens subjected to tensile strength testing were satisfactory; that is, the nature of the failures indicated that the majority of specimens failed in tension. In a few cases, however, the failure patterns were not satisfactory because they did not break at the point of smallest cross-sectional area. In

such cases, there is no assurance that failures of the test specimens were due entirely to tensile stresses; thus a more appropriate term should be used. The term "breaking tenacity," as used by the textile fiber industry (A.S.T.M. 1975) is suggested.

CONCLUSIONS

1. A statistical comparison of tensile strength values of treated sweetgum specimens exposed to *Graphium* sp. indicates no significant difference in the protective properties of 2,3,5,6,-tetrachloro-4 methylsulfonyl pyridine and pentachlorophenol.

2. Multiple comparisons of the untreated unincubated specimen mean with treated incubated specimen means indicate that 0.05 pcf of chlorosulfonyl pyridine is required to provide the same protection as afforded by 0.03 pcf of pentachlorophenol.

3. The degradation capacity of *Graphium* sp. enables this soft-rot fungus to deteriorate thin sweetgum sapwood specimens significantly during a 14-day incubation period.

4. The tensile test method as described in this study using sweetgum sapwood and *Graphium* sp. is a satisfactory procedure for evaluating the protective properties of a wood preservative.

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