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Evaluation of New Creosote Formulations after Extended Exposures in Fungal Cellar Tests and Field Plot Tests

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Abstract

This paper compares two new formulations of creosote and one pigment-emulsified creosote (PEC) with a formulation of creosote that met requirements of the AWPA standard P1/P13. Two softwood and two hardwood species were treated to four retention levels with each formulation. The evaluation of the four creosote formulations was done using (1) soil-block tests, (2) fungal cellar tests, and (3) field tests. This paper briefly discusses results from the soil-block tests, and updates previous reports to three years of fungal cellar tests and five years of field exposure in Saucier, Mississippi. Data from the latter two evaluation methods show that softwoods are generally protected better than hardwoods. Data indicate the retention is directly related to performance in both softwood and hardwood species.

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By Douglas M. Crawford, Patricia K. Lebow, and Rodney C. DeGroot

Introduction

Although creosote, or coal tar creosote, has been the choice of preservative treatment for the railroad industry since the 1920s, exuding or "bleeding" on the surface of creosote-treated products has been one incentive for further enhancements in creosote production and utility (Crawford et al., 2000). To minimize this exuding problem, laboratories such as Koppers Industries Inc., USA, and Commonwealth Scientific and Industrial Research Organization (CSIRO), Division of Chemical and Wood Technology, Melbourne, Australia, have developed changes in processing of coal tar that produce distillates with fewer contaminants. This "clean distillate" is then used to formulate "clean creosote" as a preservative.

These new, unique creosote formulations are being investigated as part of a program to enhance the use of regionally important wood species in the United States. Four retention levels of each of two new creosote formulations creosote, one pigment-emulsified creosote (PEC) and one creosote formulation that meets the AWPA Standard C2-95 for P1/P13 creosote (AWPA, 1995a), were applied to two softwood species and two hardwood species.

Two laboratory procedures, the soil-block and fungal cellar tests (accelerated field simulator), were used to evaluate the four creosote formulations. These procedures characterized the effectiveness of the wood preservatives. The soil-block tests were used to determine the minimum threshold level of the preservative necessary to inhibit decay by pure cultures of decay fungi. In general, the soil block tests showed there was little difference in the ability of the four creosote formulations to prevent decay at the three highest retention levels as summarized in a previous report by Crawford and DeGroot (1996). The soil-block tests will not be discussed in this report. Fungal cellar tests expose treated wood to mixtures of soil-borne fungi that promote accelerated attack. Crawford and DeGroot (1996) discussed the evaluation of the creosote formulations after 17 months of exposure in the USDA Forest Service, Forest Products Laboratory (FPL), fungal cellar. At that point in time data from the fungal cellar tests showed that softwoods are protected better than hardwoods for all four formulations of creosote tested. This report will discuss exposure of the fungal cellar stakes upto 36 months.

In addition, field stake tests are being used to verify service life of the new creosote formulations *in vivo*. Results from accelerated tests are indicative of field performance, but the correlation between laboratory and field results is still being investigated. Field stake tests are regarded as critical, long-term evaluations that provide results most directly related to the performance of treated products in service.

In this study, we report on the performance of the creosote formulations after five years of exposure in field tests.

Material and Methods

Wood Species

This study used the following wood species:

Douglas-fir (*Pseudotsuga menziesii*)	Mixture of heartwood and sapwood from second-growth trees in the pacific states of Oregon and Washington.
Pine (*Pinus* sp.)	Sapwood with 5 to 15 rings/inch. Wood was kiln-dried without the use of antistain chemicals.
Red oak (*Quercus rubra*)	Red oak heartwood was predominantly selected to represent a dense, ring-porous hardwood. Wood was used as supplied.
Red maple (*Acer rubrum*)	Both heartwood and sapwood of northern red maple were used to represent a diffuse porous hardwood. Wood was visually selected for clear material.

Test Specimens

Evaluations of preservative efficacy were made using the following sizes of wood specimens:

- Fungal cellar, 3 by 19 by 150 mm (0.118 by 0.748 by 5.91 in.)
- Field stakes, two sizes: 19.05 by 19.05 by 457.2 mm (0.75 by 0.75 by 18 in.) and 25 by 50 by 457 mm (1 by 2 by 18 in.).

Preservatives

The following formulations of creosote were used:

- Creosote (P1/P13) — Creosote that meets AWPA Standard P1/P13 was used as the reference preservative treatment.
- Creosote A — New formulation being developed by Koppers Industries Inc., USA.
- Creosote B — New formulation being developed by Koppers Industries Inc., USA.
- PEC — Pigment emulsified creosote—A formulation that was developed and is used in Australia (not available in USA).

Treating Process

Treating details can be found in Crawford et al. (2000). All materials were kiln-dried prior to treatment; the green Douglas-fir was kiln-dried at FPL while the other species were kiln-dried prior to arrival at FPL.

For each species, the kiln-dried materials were cut to size and equilibrated to a constant weight in accordance with procedures described in AWPA Standard E7-93 (AWPA, 1995b). Table 1 lists the various specimen sizes used in this study. Prior to treatment, stakes were sorted into groups of 30 replicates with comparable mean weight and standard deviation about the mean. Thus, each group of each species in a given size class had comparable wood densities. When these groups of 30 were treated, 10 stakes were randomly selected for analysis of treatment. The remaining 20 stakes per group were exposed in field trials.

Table 1. Sizes and exposure location of specimens.

Specimen size		Exposure
(mm)	(in.)	Saucier, MS; FPL
25 by 50 by 500	1 by 2 by 20	Field plots
19 by 19 by 457	0.75 by 0.75 by 18	Field plots
3 by 19 by 150	0.118 by 0.75 by 5.91	Fungal cellar (FPL)

The sorted, equilibrated wood materials to be treated with PEC were shipped to CSIRO in Australia. All other treatments with the two new formulations of creosote and reference AWPA P1/P13 creosote were performed at FPL. Within each formulation of creosote, four concentrations (65%, 30%, 15% and 7.5% active ingredient (ai)) were used to treat the wood samples. The PEC creosote was diluted with water; other formulations were diluted with toluene. Each wood species was treated with the same set of four treating solutions. This produced a series of creosote retention levels within each wood species, but the actual retention levels resulting from treatment with any given concentration of treating solution varied among species. Actual retention within the respective individual stakes was determined on the basis of weight gain after treatment.

Fungal Cellar

Fifteen replicate stakes of each wood species were exposed for each of the four retention levels of each creosote formulation. Soil beds were maintained in a controlled environment at 26°C (79°F) and a relative humidity of 86% to 90%. The soil is maintained at moisture content of 50% to 70% of water holding capacity to promote growth of soft-rot fungi (Nicholas et al., 1991). Prior to exposure in the fungal cellar all treated and control specimens were vacuum impregnated with water. The stakes were grouped by treatment, subjected to vacuum at –13.0 kPa (about 100 mmHg) for 30 minutes before being soaked for 2 hours in distilled water. The test specimens were then inserted vertically into the soil bed until the top end was level with the soil.

At 3, 6, 9, 12, 17, and 36 months (30.4 days/mo), wood specimens were removed from the fungal cellar, cleaned with a brush to remove excess soil, and placed in watertight plastic bags until evaluation for strength loss using the bending strength apparatus. Strength loss was determined as described by Crawford (1994). Care was taken to ensure that the test specimen was oriented in the same way during subsequent strength evaluations. After each strength evaluation, specimens were returned for exposure in the soil beds.

Initial load measurements were made, and modulus of elasticity (MOE) was calculated. The MOE was calculated using Equation (1).

$$MOE = \frac{L^3}{4} \frac{P}{bh^3} \frac{}{D} \quad = 389.94(P) \qquad (1)$$

where

MOE = modulus of elasticity (kPa), L = constant span of 126 mm, b = constant specimen width of 19 mm, h = constant specimen thickness of 3 mm, D = constant specimen deflection of 2.50 mm and P = variable force to maintain constant deflection (g).

Stakes that demonstrated MOE losses greater than 60% of their original MOE were considered failures but remained in the fungal cellar with periodic strength evaluations. Only broken stakes were removed.

Field Plots

Stakes have been exposed in ground contact for five years in plots at Saucier, Mississippi. The stake tests involved a minimum of four retentions for each preservative/species/site combination. Twenty replicate stakes were used for each variable. Table 2 gives the grading system for the in-ground stake evaluations. The Saucier field plot site located on the Harrison Experimental Forest in Mississippi has a mean annual precipitation 1,580 mm (62.2 in.) and an average annual temperature 19.6°C (67.3°F). The soil type is poarch sandy loam.

Table 2. The grading system used to determine the index of condition for stakes in ground. (1)

Decay grades (index of condition)	
Grade No.	Description of condition
10	Sound. Suspicion of decay permitted
9	Trace decay to 3% of cross section
8	Decay from 3% to 10% cross section
7	Decay from 10% to 30% cross section
6	Decay from 30% to 50% cross section
4	Decay from 50% to 75% cross section
0	Failure

(1) AWPA (1995b).

Results and Discussion

Fungal Cellar Test

The treatability of all four wood species at a given concentration of active ingredient of the different formulations provided a range in retention levels of active ingredient in the treated wood that ranged from slightly more than 16 kg/m^3 (1 pound per cubic foot (pcf)) to approximately 320 kg/m^3 (20 pcf) (Table 3). This range in retention of active ingredient spanned the targeted retention of 160 kg/m^3 (10 pcf) that is specified for oak ties (AWPA, 1995b).

Table 3—Average retention at different concentrations of four creosote formulations in fungal cellar test.

Formulation	Concen-tration (% ai[a])	Pine (pcf)	Pine (kg/m^3)	Douglas-fir (pcf)	Douglas-fir (kg/m^3)	Red oak (pcf)	Red oak (kg/m^3)	Red maple (pcf)	Red maple (kg/m^3)
PEC[b]	7.5	2.99	47.92	3.33	53.37	2.94	47.12	3.12	50.00
	15	5.76	92.31	5.76	92.31	5.55	88.94	5.36	85.90
	30	12.98	208.01	12.99	208.17	12.68	203.21	12.64	202.56
	65	27.75	444.71	23.11	370.35	19.06	305.45	23.35	374.20
P1/P13	7.5	2.07	33.17	1.99	31.89	1.20	19.23	1.65	26.44
Creosote	15	4.15	66.51	4.09	65.54	2.45	39.26	3.52	56.41
	30	11.15	178.69	9.72	155.77	10.68	171.15	9.52	152.56
	65	20.60	330.13	20.55	329.33	14.24	228.21	18.12	290.38
Creosote A	7.5	1.98	31.73	1.79	28.69	1.17	18.75	1.63	26.12
	15	4.16	66.67	3.67	58.81	2.51	40.22	3.66	58.65
	30	8.90	142.63	8.50	136.22	5.47	87.66	7.96	127.56
	65	20.15	322.92	20.07	321.63	20.60	330.13	13.19	211.38
Creosote B	7.5	1.96	31.41	2.02	32.27	1.24	19.87	1.87	29.97
	15	4.04	64.74	1.94	31.09	2.43	38.94	1.89	30.29
	30	8.89	142.47	7.63	122.28	5.56	89.10	8.23	131.89
	65	21.35	342.15	20.52	328.85	13.28	212.82	20.24	324.36

[a]ai is active ingredient.
[b]PEC is pigment emulsified creosote.

At higher concentrations, retention levels for creosote P1/P13, A and B were approximately 20% less than that achieved with PEC. Still, these retention levels were greater than 160 kg/m^3 (10 pcf).

Red oak had the lowest retention at most solution concentrations for all formulations. The PEC yielded the highest retentions as calculated by weight gain. Retention levels of the other three formulations tended to be relatively comparable for each concentration of treating solution, but with an occasional spurious result for a given wood species.

Modulus of elasticity (MOE) results are reported for 36 months of exposure (Table 4). None of the creosote formulations appeared to prevent attack by soft-rot fungi in red oak or red maple as determined by reduced MOE, although treatments at higher retentions resulted in losses. Decrease in

MOE exceeded 60% at all retention levels for all creosote formulations in those species. At the higher creosote retention levels, softwoods remained better protected than hardwoods.

Table 4—Median and Mean of Modulus of elasticity loss (%), number of broken specimens and number of failed specimens at 156 weeks of exposure in fungal cellar test.

	Modulus of elasticity loss (%)					
	Concentration (% ai[a])	Creosote (P1/P13)	Creosote A	Creosote B	PEC[a]	Control
Pine	0					100.00 100.00 25/25
	7.5	93.7 93.0 1/15	95.1 93.4 6/15	97.6 96.2 7/15	97.8 96.7 7/15	
	15	84.5 83.7 0/15	93.1 91.5 2/15	90.8 90.0 2/15	94.8 93.8 2/15	
	30	76.8 75.8 0/15	80.7 80.0 1/14	81.3 77.1 1/13	81.3 80.9 1/15	
	65	41.6 40.4 0/0	54.7 54.9 0/6	45.8 43.9 0/1	27.6 28.2 0/0	
Doug-fir	0					100 99.4 22/25
	7.5	92.0 90.8 2/15	92.5 93.9 5/15	98.3 95.0 7/15	100.0 99.5 14/15	
	15	80.4 80.7 1/15	84.4 83.6 1/14	84.5 84.7 3/15	100.0 98.8 13/15	
	30	65.5 65.8 0/12	61.2 61.9 0/9	66.2 65.2 0/10	93.1 89.9 6/14	
	65	27.3 34.8 1/1	38.9 38.3 0/1	42.7 41.8 0/1	25.2 24.9 0/1	
Red oak	0					100.0 100.0 25/25
	7.5	100.0 97.6 14/15	100.0 100.0 15/15	100.0 100.0 15/15	100.0 99.7 13/15	
	15	100.0	100.0	100.0	98.2	

	98.7	99.2	99.5	98.5
	9/15	10/15	12/15	6/15
30	93.7	96.5	100.0	96.6
	93.7	96.6	99.2	96.2
	0/15	1/15	12/15	3/15
65	84.6	89.7	91.1	82.5
	84.9	90.2	91.0	84.0
	1/15	1/15	0/15	0/15

Table 4 (cont.)—Median and Mean of Modulus of elasticity loss (%), number of broken specimens and number of failed specimens at 156 weeks of exposure in fungal cellar test.

	Modulus of elasticity loss (%)					
	Concentration (% ai[a])	Creosote (P1/P13)	Creosote A	Creosote B	PEC[a]	Control
Red maple	0					100.0 100.0 25/25
	7.5	100.0 100.0 15/15	100.0 100.0 15/15	100.0 100.0 15/15	100.0 100.0 15/15	
	15	100.0 98.9 9/15	100.0 100.0 15/15	100.0 99.3 11/15	100.0 99.8 13/15	
	30	91.0 90.8 0/15	97.3 97.5 6/15	97.6 97.2 7/15	100.0 98.9 10/15	
	65	80.6 79.9 0/15	87.2 83.2 0/15	81.8 83.9 0/15	80.7 81.4 0/15	

[a]PEC is pigment emulsified creosote.

Marginal losses in MOE were observed in Douglas-fir stakes and southern pine stakes treated with 65% PEC after 36 months of exposure in the fungal cellar. Both hardwood species showed approximately 80 to 90% loss in MOE at 65% PEC after 36 months of exposure in the fungal cellar. Loss in MOE for other formulations in Douglas-fir ranged from about 60% to 100% at active ingredient concentration of 7.5% to 30%.

With the exception of treatments at the high retention levels, degradation of residual strength of each stake can be reasonably modeled using the logarithmic transform followed by linear regression. Thus, for each stake, a residual strength loss rate is obtained. These rates can then be used for treatment comparisons, as well as for further model exploration. Figure 1 shows the average rates (in terms of percentage loss per month) for each species, treatment, and retention combination versus the actual retention levels (on log scale). Comparison of the rates within each species, via an unweighted ANOVA followed by Scheffe's mean comparison procedure, confirmed some protection, compared with the untreated controls, in all of the species, and increasing protection with increasing retentions. The exceptions were red oak at the low retention levels and PEC-treated Douglas-fir at the low retention levels.

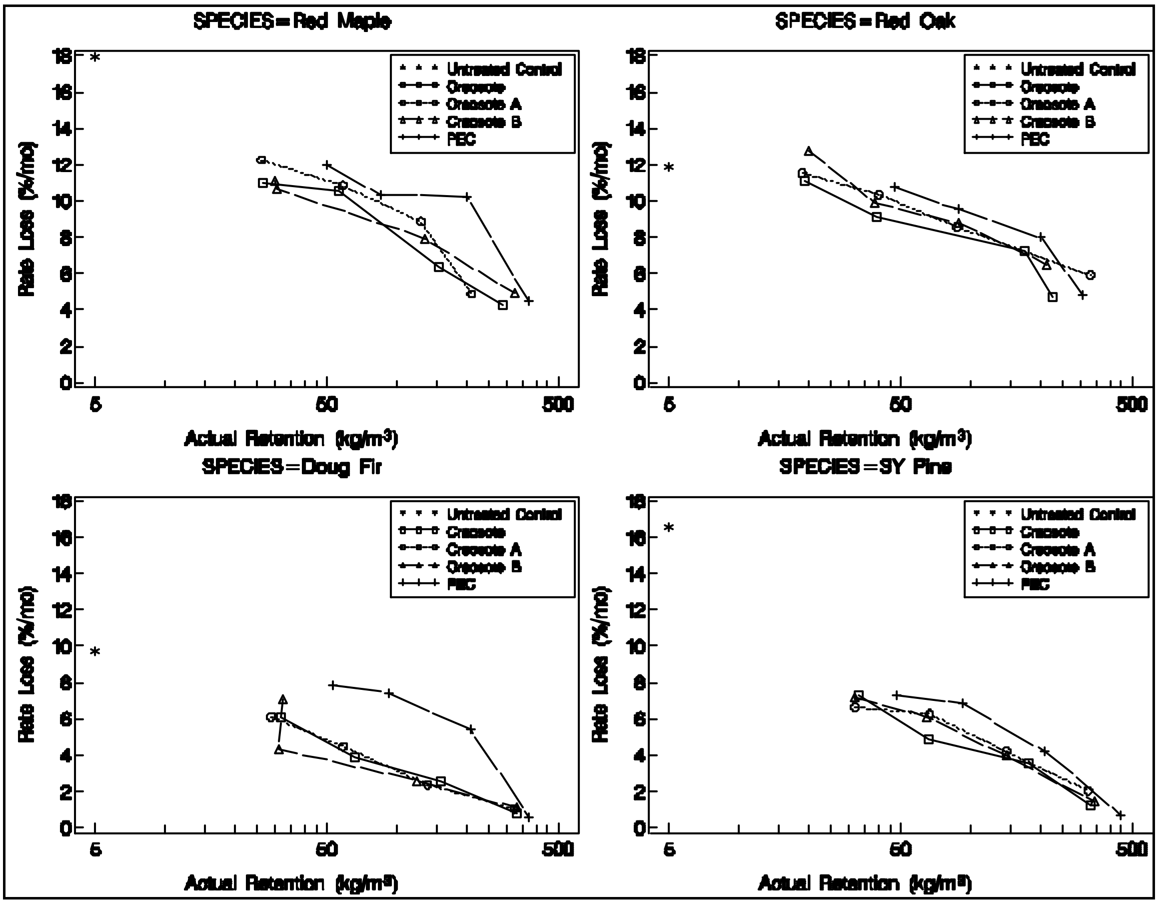

Figure 1. Plot of fungal cellar strength loss rates (%/mo) versus actual retentions for each species. Note that untreated controls are plotted at retention 5 for display purposes.

Comparison of formulation effectiveness is difficult because of the different retentions levels achieved with each formulation. However, general trends indicate relatively little difference in effectiveness between the four formulations.

Field Plot Results

For the field plot specimens, the treatability of all four wood species at a given concentration of active ingredient of the different formulations provided a range in retention levels of active ingredient (see Tables 5 and 6 for actual retentions).

Table 5—Average retention at different concentrations of four creosote formulations in 19-mm (3/4-in.) stakes in field plots.

Formulation	Concen-tration (% ai[a])	Pine (pcf)	Pine (kg/m^3)	Douglas-fir (pcf)	Douglas-fir (kg/m^3)	Red oak (pcf)	Red oak (kg/m^3)	Red maple (pcf)	Red maple (kg/m^3)
PEC[b]	7.5	2.90	46.35	3.02	48.25	2.76	44.15	2.59	41.40
	15	5.75	91.94	5.99	95.82	5.49	87.78	5.41	86.60
	30	11.44	183.05	11.42	182.79	10.18	162.95	9.41	150.60
	65	26.08	417.32	24.71	395.28	16.31	261.03	19.59	313.50
P1/P13 creosote	7.5	2.30	36.74	2.19	35.04	1.62	25.93	1.98	31.76
	15	4.59	73.42	4.55	72.85	3.23	51.74	4.01	64.18
	30	9.56	152.95	9.80	156.85	6.76	108.10	8.22	131.50
	65	22.34	357.42	22.03	352.48	16.02	256.40	19.69	315.09
Creosote A	7.5	2.26	36.19	2.28	36.42	1.56	25.02	1.96	31.42
	15	4.57	73.14	4.74	75.82	3.29	52.65	3.99	63.90
	30	9.36	149.80	9.45	151.19	6.71	107.29	8.39	134.31
	65	22.25	355.96	21.79	348.60	15.67	250.76	19.81	317.01
Creosote B	7.5	2.26	36.18	2.26	36.20	1.63	26.10	2.02	32.35
	15	4.53	72.42	4.72	75.55	3.23	51.66	4.01	64.22
	30	9.28	148.41	9.23	147.64	6.61	105.80	8.09	129.40
	65	22.27	356.24	21.08	337.34	15.37	245.91	18.55	296.84

[a]ai is active ingredient.
[b]PEC is pigment emulsified creosote.

Table 6—Average retention at different concentrations of two creosote formulations in 25- by 50-mm (1- by 2-in.) stakes in field plots.

Formulation	Concen-tration (% ai[a])	Pine (pcf)	Pine (kg/m^3)	Douglas-fir (pcf)	Douglas-fir (kg/m^3)	Red oak (pcf)	Red oak (kg/m^3)	Red maple (pcf)	Red maple (kg/m^3)
PEC[b]	7.5	2.23	35.67	2.87	45.88	2.29	38.80	-	-
	15	5.05	80.87	5.95	95.26	4.90	78.37	-	-
	30	8.75	139.98	9.86	157.75	7.25	115.96	6.64	106.32
	65	23.46	375.37	24.06	385.00	14.19	227.01	19.24	307.83
P1/P13 creosote	7.5	2.23	35.75	2.21	35.58	1.79	28.58	-	-
	15	4.51	72.18	4.63	74.03	3.64	58.20	-	-
	30	9.38	150.11	9.25	147.98	7.39	118.30	-	-
	65	21.98	351.73	22.43	358.82	17.60	281.57	-	-

[a]ai is active ingredient.
[b]PEC is pigment emulsified creosote.

For each treatment group, the average visual index of condition of the 19-mm (3/4-in.) stakes exposed in Mississippi are shown in Figure 2; similarly the 25 by 50 mm (1 by 2 in.) results are shown in Figure 3. Within each species, the average visual ratings are ranked in descending order; those with equal rankings are ordered alphanumerically (descending). After five years exposure in the field, treated stakes started showing signs of visual degradation at lower retention levels.

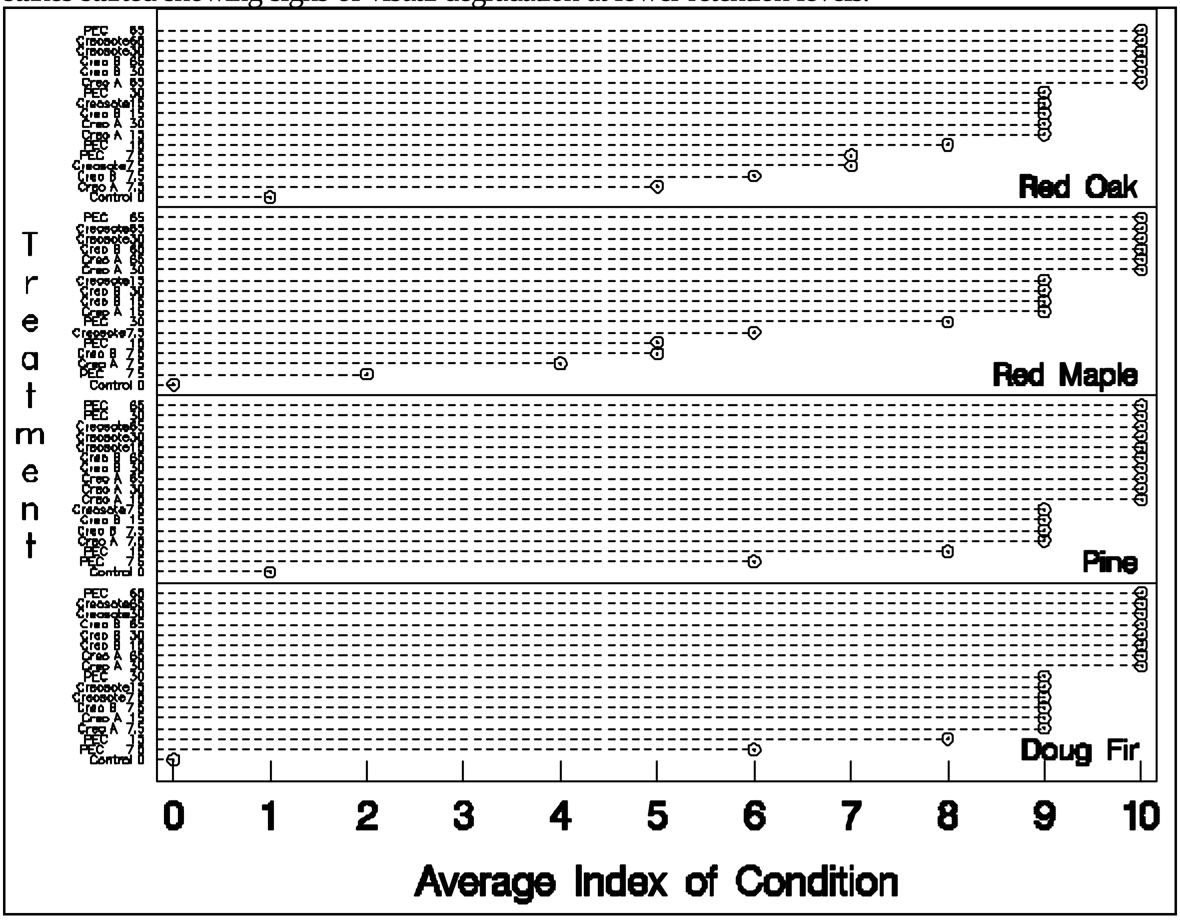

Figure 2. Average index of condition for the 19-mm (3/4-in.) stakes exposed for five years in Mississippi.

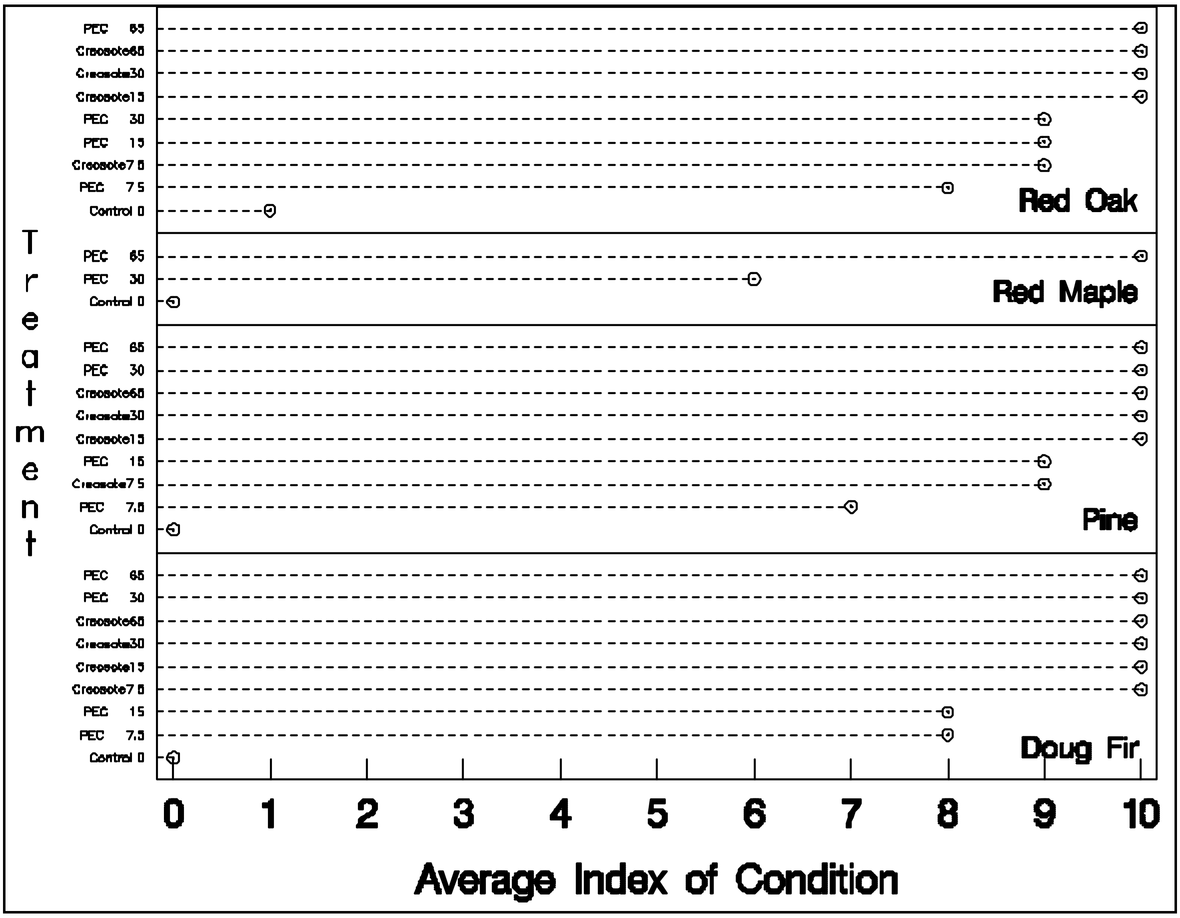

Figure 3. Average index of condition for the 25 x 50 mm (1 x 2 in) stakes exposed for five years in Mississippi.

Ideally the fungal cellar test results would be indicative of performance in the field. A measure of association between field performance, as measured by the average visual index of condition, and the fungal cellar, as measured by the negative of the estimated degradation rates, is possible with nonparametric tests of association. These tests have similar interpretations as tests based on Pearson's correlation coefficient between continuous variates but provide information about relationships if one of the variates is only measured on an ordinal scale. The first test of association is based on the coefficient of rank correlation (also called Spearman's Rho) and is basically the usual correlation between the ranks of the two variates. The second test of association is called Kendall's Tau coefficient and is a measure of the number of concordant pairs (that is, pairs in which the direction of rank ordering for each variate of the pairs is the same for both variates) versus the number of discordant pairs (that is, the direction of rank orderings between the two variates is not the same). In general, both statistics fall between -1 and 1, with values close to -1 indicating high negative association and values close to 1 indicating high positive association. Values close to zero indicate lack of an association. The p-values for the tests are the probability that, given no association, of calculating at least that association value. See Gibbons (1985) for further details. These tests will at least give some indication that the order of performance, as based on the above measures, is similar between the two exposure environments.

Table 6 gives the measures of association within each species between the average visual ratings of the field stakes and the negative of the average strength loss rates from the fungal cellar stakes. The two values are not directly comparable because Kendall's Tau coefficient is typically less than Spearman's Rho; however, the p-values for the tests of association based on these values are comparable. For the 19-mm (3/4-in.) stakes, all association measures had significant p-values ≤ 0.0001 with the exception of Kendall's Tau for southern pine, which had a p-value of 0.0003. Measures for the 25- by 50-mm (1- by 2-in.) stakes were similar but had reduced p-values. Thus, there is significant evidence that the two measures are associated. More work is needed to exploit this relationship, but this positive association plus the linearity of degradation rates encourage further model development.

Table 6. Association analysis between the negative of strength degradation rates from accelerated tests and the index of condition from field tests of 19-mm stakes (each species group N=17).

Species	Spearman's Rho (p-value)	Kendall's Tau (p-value)
Douglas-fir	0.91(0.0001)	0.81(0.0001)
Southern pine	0.84(0.0001)	0.72(0.0003)
Red maple	0.90(0.0001)	0.77(0.0001)
Red oak	0.91(0.0001)	0.80(0.0001)

Concluding Remarks

Results from the soil-block test indicate that products treated with any formulation in the test should have comparable durability. Fungal cellar tests have shown the same trends as the soil blocks. However, results to date from the fungal cellar tests indicate the potential for poorer performance of treated hardwoods than has been observed in practice. The historical success of P1/P13-type creosotes in U.S. hardwoods at retention levels less than those tested in this study begs for a fundamental explanation of the cause of these results.

The relative low retention of red oak in comparison with retention levels of other formulations per concentration of treating solution may somehow be related to the relatively poor performance of that wood species in the fungal cellar. Still, a calculated retention in excess of 160 kg/m^3 (10 pcf) was obtained with all formulations at the highest treatment concentration. Furthermore, red maple, which usually had higher retention levels than red oak for each treatment, also performed poorly in the fungal cellar.

PEC was retained at a higher loading level than the other formulations. This could mean that a treatment could be developed that has the same efficacy at the lower loading level as other creosote formulations.

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