TREATABILITY OF U.S. WOOD SPECIES WITH PIGMENT-EMULSIFIED CREOSOTE

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ABSTRACT

Since the 1920s creosote has been used extensively in the United States for treatment of construction timbers, poles, and posts. However, creosote has the tendency to exude or "bleed" from some treated commodities, producing a tar-like covered surface. In the United States, creosote-treated products exhibiting cleaner dried surfaces and a reduced tendency to bleed have been achieved through reduction of the xylene-insoluble carbonaceous fraction in creosote. In Australia, pigment-stabilized creosote emulsion formulations have been designed and developed to "1ock"the oil phase within the treated timber and are referred to as pigment-emulsified creosote (PEC). The surfaces of PEC-treated commodities remain dry; the creosote does not leach into the ground or water in marine environments; and the oil remains mobile within the microstructure of the PEC-treated products. In this study, the treatment characteristics of southern pine, red oak, red maple, and Douglas-fir with PEC 30W are reported. Results showed that treatment of the four wood species with PEC 30W is generally comparable to treatment with reference creosote Pl/P13, except that slightly greater variability in creosote loading occurs with PEC.

A recent survey of U.S. engineers interested in transportation structures revealed a high priority need for new preservatives to treat wood species used in bridge construction (20). In response to that need, a program was initiated at the USDA Forest Service, Forest Products Laboratory (FPL), Madison, Wis., to investigate new preservatives that could contribute to enhanced utilization of regionally important wood species in the United States (6). New andunique formulations based upon creosote are included in the program (5).

Creosote has been used as a wood preservative for more than 180 years. The use of creosote as a wood preservative in the United States began around 1870 when a pressure-treatment plant was constructed in Pascagoula, Miss., to produce treated railroad ties. By the 1920s creosote was the treatment of choice for the railroad industry and continues to be so today (7). Creosote is also used extensively within the United States for treatment of construction timbers, poles, and posts. On a worldwide basis, creosote is the highest volume usage wood preservative (11). In the United States, approximately 15 percent of the total volume of wood treated with wood preservatives is treated with creosote (2).

The tendency of creosote to exude or 'bleed" from some treated commodities, producing an oily or tar-like ("crud") covered surface (12), can cause handling problems and has increased public concern about its effect on the environment. Creosote bleeding from the surfaces of its treated products contributes to a reduction in public acceptance of creosote-treated products. Ongoing research on formulation and processing steps has addressed concerns about appearance, smell, and handling characteristics of creosote-treated products.

In the United States, creosote-treated products exhibiting cleaner dried surfaces and a reduced tendency to bleed

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TABLE 1. – Wood species used in this study.

Common name	Scientific name	Description and source
Douglas-fir	*Pseudotsuga menziesii* (Mirb.) Franco	Mill-run wood from second-growth trees in the Pacific Coastal region of Oregon. Wood furnish was mostly heartwood, but some sapwood was present. Small amounts of sapwood (less than 15% of cross-sectional area) occurred in some wood members.
Red oak	*Quercus rubra* L.	Heartwood of northern red oak from Wisconsin
Red maple	*Acer rubrum* L.	Mill-run wood from northeastern United States. No distinction was made between heartwood and sapwood, but furnish appeared to be mostly sapwood.
Southern pine	*Pinus elliotii, P. palustris,* or *P. taeda*	Sapwood with 5 to 15 rings per 25 mm (1 in.) from trees in Alabama. Exact species of trees harvested was not determined, but these species predominate in the area of harvest.

TABLE 2. – General specifications for PEC 30W (8).

High temperature creosote content:	65.1% w/w > 2% (A.S. 1143)
Water phase components:	30.0% w/w > 2% (includes up to 0.7% m/m soluble pigment dispersion base and 3.0% m/m other soluble components)
Predispersed sub-micro pigment content:	3.5% w/w (non-water soluble pigment components 2% to 8% m/m) minimum (solids content)
Non-distillable components:	1.8% w/w (surfactants and other additives)
pH:	9 to 11
Density at 20°C:	1.08 minimum
Typical apparent viscosity (50°C 210 s-l):	11 to 21 mPa.s
Surface tension 20°C:	35 m N maximum

TABLE 3. – Sizes of wood specimens that were treated.

Sample size		Exposure
(mm)	(in.)	
25 by 50 by 500[a]	1 by 2 by 20	Field plots (Australia and USA)
19 by 19 by 457[b]	0.75 by 0.75 by 18	Field plots (USA)
19 by 19 by 19[b]	0.75 by 0.75 by 0.75	Soil bottles (FPL)
3 by 19 by 150[b]	0.118 by 0.75 by 5.91	Fungal cellar (FPL)

[a] Only stakes of this size included in this report on treatability.
[b] Treated and used in additional efficacy studies.

have been achieved through reduction of the xylene-insoluble carbonaceous fraction in creosote (19). In Australia, pigment-stabilized creosote emulsion formulations have been designed and developed to "lock" the oil phase within the treated timber by the unique action of the submicro pigment, which is dispersed through both phases of the emulsion but is mainly partitioned at the water-oil interface (10,14). This preservative composition is referred to as pigment-emulsified creosote (PEC). Important features of PEC-treated wood are that it exhibits dry, oil-free surfaces (10), and the surfaces of PEC-treated commodities remain dry. The oil remains mobile within the microstructure of the PEC-treated products. Emulsification of creosote obviates crystallization of high boiling fractions, which are for the first time uniformly pressure impregnated into the PEC-treated commodities for enhanced efficacy. PEC can be utilized at a considerably lower temperature than creosote to achieve the same treatment results.

In this study, we report our initial observations of four U.S. species treated with PEC 30W, which is an anionic emulsion of creosote with 30 percent water and contains a dispersed submicro titanium dioxide pigment (17). Extensive research and experiments with PEC in Australia used primarily the eucalyptus species (3,9). However, additional significant research and pilot-scale testing of PEC 30B and PEC 30W to treat U.K. timbers, Scandinavian timbers, Malaysian timbers, and German timbers has been successfully carried out (4,18).

To gain insight into the potential for treating U.S. wood species with PEC 30W, a cooperative study was initiated in collaboration with Commonwealth Scientific and Industrial Research Organization (CSIRO), Division of Forest Products, Clayton Victoria, Australia (now called CSIRO Forestry and Forest Products).

The long-term objective of this study is to evaluate the durability of four U.S. wood species treated with PEC 30W. Characterization and assessment of treatment of these wood species is the initial step of this evaluation. This study reports results only on treatability of stakes 25 by 50 mm (1 by 2 in.) in cross section. With stakes of this dimension, both laboratories independently evaluated results of treatment with PEC 30W. Analysis of treatment with the reference creosote was done only at FPL.

For efficacy studies, we also treated stakes of smaller dimension: 19 by 19 by 457 mm (0.75 by 0.75 by 18 in.), 3 by 19 by 150 mm (0.12 by 0.75 by 5.91 in.), and 19 by 19 by 19 mm (0.75 by 0.75 by 0.75 in.). Results from efficacy studies are not included in this report.

MATERIALS AND METHODS

In this study, the treatability and subsequent efficacy of PEC 30W were compared with that of reference P1/P13 creosote in four U.S. wood species **(Table 1).** The reference creosote formulation meets the requirements of American Wood-Preservers' Association (AWPA) Standard P1/13-95 (1). General specifications for a typical white PEC having a 30 percent water content (PEC 30W) are listed in **Table 2** (8).

The southern pine, red oak, and red maple material was kiln-dried prior to acquisition by FPL. Upon arrival at FPL, the green Douglas-fir lumber was kiln-

dried following a mild schedule that began with a dry-bulb temperature of 43.3°C (110°F) and concluded with 71.1°C (160°F), with a -13.33°C (8°F) depression between wet- and dry-bulb throughout. The dried material was then cut into long strips either 19 by 19 mm or 25 by 50 mm in cross section **(Table 3).** These lengths were then subjected to a vacuum of -94.82 kPa (28 in.) Hg for 15 minutes, then flooded with water and allowed to stand overnight at atmospheric pressure. The 25- by 50-mm members were incised on the two broad faces to a depth of 8 mm (0.32 in.), with an experimental knife incisor that produced approximately 130 incisions per 93 by 10^{-3} mm^2. The 19- by 19-mm members were incised to a depth of 5 mm on the radial surfaces only. Following incising, the wood members were again kiln-dried using a schedule that began with an initial dry-bulb temperature of 47.6°C (120°F) and concluded with a dry-bulb temperature of 65.5°C (150°F). A 10°F depression between dry- and wet-bulb temperatures was maintained throughout the schedule.

The selection process for stakes was designed to yield groups of 30 replicated stakes with comparable mean weight and distribution of weights for each stake size within each respective species. This was accomplished 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 E7-93 (1). Within each size group for each species, equilibrated stakes were then weighed and ordered according to weight. 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.

The sorted, equilibrated wood materials to be treated with PEC were shipped to CSIRO in Australia. All treatments with the reference AWPA PUP13 creosote were performed at FPL. With each formulation of creosote, only four concentrations were used to treat all woods **(Table 4).** Each wood species was treated

TABLE 4. *– Concentration levels of creosote in treating solutions.* [a]

Reference creosote (AWPA P1/P13)	Pigment-emulsified creosote (PEC 30W)
----------(%)----------	
65	65
30	30
15	15
7.5	7.5

[a] Concentration shown is the actual percentage of creosote in the emulsion treating solution.

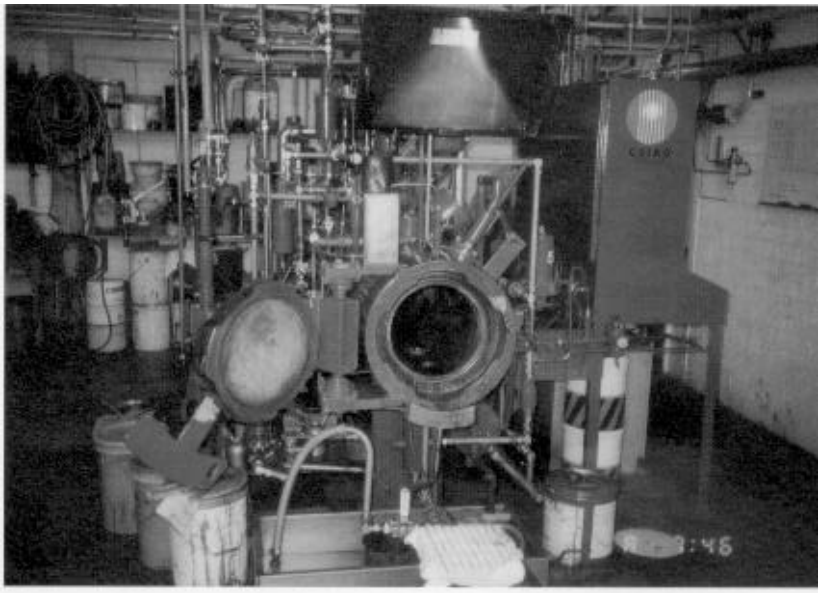

Figure 1. – Treating cylinder at CSIRO.

TABLE 5. *– Creosote retention in sets of 30 stakes as determined by weight gain.*

Creosote[a]	Concentration	Lab set[b]	Douglas-fir		Red maple		Red oak		Southern pine	
	(%)		(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)
PEC	65	CSIRO	385 (44)[c]	24 (3)	307 (70)	19 (4)	227 (35)	14 (2)	375 (32)	23 (2)
PEC	65	FPL	389 (437	24 (3)	319 (82)	20 (5)	226 (39)	14 (2)	374 (31)	23 (2)
P1/P13	65	FPL	359 (31)	22(2)	--[d]	--	282 (16)	18 (1)	352 (22)	22 (1)
PEC	30	CSIRO	158 (44)	10 (3)	106 (42)	7 (3)	116 (36)	7 (2)	140 (18)	8 (1)
PEC	30	FPL	167 (32)	10 (2)	112 (43)	7 (3)	140 (29)	9 (2)	134 (23)	8 (1)
P1/P13	30	FPL	148 (17)	9 (l)	--	--	118(8)	7 (0.5)	150 (8)	9 (0.5)
PEC	15	CSIRO	95 (5)	6 (0.3)	--	--	78 (7)	5 (0.4)	81 (7)	5 (0.4)
PEC	15	FPL	84 (27)	5 (2)	--	--	77 (5)	5 (0.3)	80 (7)	5 (0.5)
P1/13	15	FPL	74 (7)	5 (0.4)	--	--	58 (3)	4 (0.2)	72 (4)	5 (0.3)
PEC	7.5	CSIRO	46 (2)	3 (0.1)	--	--	39 (2)	2 (0.1)	36 (2)	2 (0.1)
PEC	7.5	FPL	49 (3)	3 (0.2)	--	--	38 (8)	2 (0.5)	36 (2)	2 (0.1)
P1/P13	7.5	FPL	35 (3)	2 (0.2)	--	--	29 (2)	2 (0.1)	36 (2)	2 (0.1)

[a] Two sets of 30 stakes were treated with PEC W30 at CSIRO. One set of 30 stakes was treated with P1/P13 at FPL.
[b] Sets of 30 stakes are identified by the ultimate field location in which they were exposed.
[c] Values in parentheses are standard deviations.
[d] Not treated.

TABLE 6. – *Comparison of creosote retention in PEC-treated stakes as determined by weight gain at CSIRO and independently by chemical analysis at CSIRO and FPL.* [a]

Timber species, % creosote	CSIRO creosote weight gain		CSIRO extraction		FPL creosote weight gain		FPL extraction	
	(kg/m^3)	(pcf)	(kg/m^3)	(pcf)	(kg/m^3)	(pcf)	(kg/m^3)	(pcf)
Douglas-fir								
65	361	22	304	18	386	24	414	25
30	162	10	89	6	172	11	85	5
15	95	6	48	3	94	6	49	3
7.5	46	3	24	2	49	3	34	2
Southern pine								
65	375	23	392	24	370	23	446	28
30	136	8	72	4	127	8	76	5
15	81	5	48	3	83	5	66	4
7.5	36	2	33	2	36	2	46	3
Red maple								
65	291	18	270	16	310	19	337	21
30	106	7	41	3	124	8	25	2
15								
7.5								
Red oak								
65	227	14	181	11	216	13	90	6
30	116	7	74	7	153	9	62	4
15	78	5	57	3	77	5		
7.5	39	2	26	2	38	2	22	1

[a] Each datum represents the average of 10 replicate stakes randomly selected from sets of 30 stakes per wood species by treatment combination.

TABLE 7. – *Comparison of creosote retention levels in sets of 10 P1/P13-treated stakes as determined by weight gain and chemical extraction at FPL.*

Timber species, % creosote	Weight gain		Extraction	
	(kg/m^3)	(pcf)	(kg/m^3)	(pcf)
Douglas-fir				
65	354	22	343	21
30	146	9	220	14
15	74	5	62	4
7.5	35	2	13	0.8
Southern pine				
65	353	22	419	26
30	150	9	163	10
15	71	4	96	6
7.5	36	2	30	2
Red oak				
65	285	18	229	14
30	118	7	71	4
15	60	4	6	0.4
7.5	28	2	-1.8	-0.1

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 during treatment, each specimen being weighed immediately before and after treatment, and on chemical analysis after treatment.

All treatments with PEC were carried out at the Clayton Forest Products Pilot Plant of CSIRO **(Fig. 1)** by a process that utilizes an apparatus (15) for ultra high shear emulsification of the hydrophobic and hydrophilic phases of the treating solution. Stakes of each wood species were treated separately. Douglas-fir, red oak, and southern pine stakes of both size classes were treated with one of four concentrations of PEC. Red maple stakes were treated with two concentrations. At each treatment level, 2 sets of 30 25- by 50-mm (1- by 2-in.) stakes were treated. One set of the 25- by 50-mm (l- by 2-in.) stakes was retained in Australia and the other returned to the United States. Thus, chemical analyses at the individual laboratories were conducted on different but simultaneously treated sets of stakes. Results of treatment are presented separately for both sets to provide indications of uniformity in treatment, as determined by weight gain, and the analytical results at the respective laboratories compared with weight gain determinations for their respective sets.

All treatments with the reference creosote were carried out at FPL using a modified full-cell process, with an initial vacuum of at -94.82 kPa (28 in.) Hg for 30 minutes and press time of 180 minutes at 1,005 kPa (150 psi); a final vacuum was applied for 30 minutes. Each treating solution was prepared by diluting creosote with toluene on a w/w percent basis. At each concentration of reference creosote, Douglas-fir and red oak samples of comparable size were simultaneously treated. Similarly, southern pine and red maple samples of comparable size were treated simultaneously at each solution concentration. At the conclusion of the respective treating cycle, samples were removed, blotted dry, and weighed.

Treatability was evaluated on the basis of weight gain in all 30 stakes during

treatment and on chemical analysis an observed penetration and distribution in 10 stakes per group. Immediately upon removal from the treatment cylinder, stakes were blotted dry and weighed to determine weight gain. The retention of creosote per stake as determined by this method is expressed on a weight-to-volume basis **(Table 5).**

From each set of 30 stakes, 10 stakes were reserved for chemical analysis **(Tables 6** and **7)** and measurement of penetration. After air-drying for approximately 1 year at each laboratory, cross sections were cut from midlength of each of the 10 stakes per set of 30 for creosote extraction. Two lo-mm-thick cross sections were solvent extracted at CSIRO; five 6-mm-thick cross sections were extracted at FPL. Extraction procedures were in accordance with those described in AWPA A6-97 (1).

At FPL, the cross section face at midlength of each of 10 stakes per treatment-by-retention-by-wood combination was visually inspected to determine depth of penetration. The percentage of cross section that was penetrated was determined for each stake **(Table 8).** For each of the 10 stakes, we recorded whether 0 to 25 percent, 26 to 50 percent, 51 to 75 percent, or 76 to 100 percent of the cross sectional area had been penetrated by creosote. Following the procedure of method 2 of AWPA M2-97 (1), annual rings were considered penetrated if any portion of that ring was penetrated. Thus, a positive result was penetration of both earlywood and latewood as well as just the latewood **(Figs. 2** through **4).** A quantitative comparison of treatments was accomplished by analyzing the amount of creosote in the outer 6 mm and in the central core of five stakes or species that were treated with both formulations at the solution and emulsion concentration of 65 percent creosote **(Table 9).**

RESULTS

Average retention levels with the series of creosote concentrations, as determined by weight gain in sets of 30 stakes during treatment **(Table 5),** were similar for both formulations of creosote in southern pine, red oak, and Douglas-fir, but the standard deviation for populations of 30 stakes was greater with PEC W30 than with P1/P13 creosote. Results from chemical analysis **(Tables 6** and **7)** are based on the sets of 10 stakes (ran-

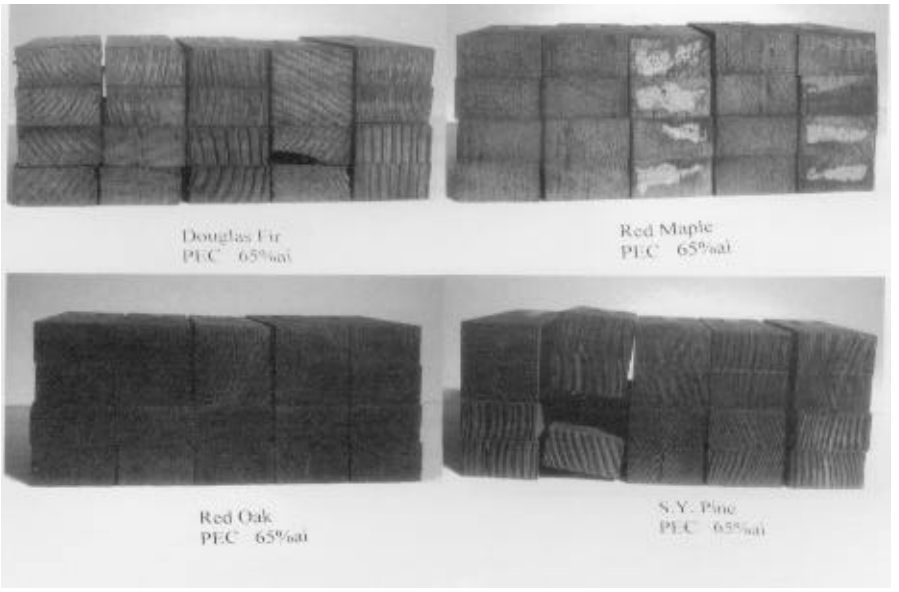

Figure 2. – Cross section of 10 stakes of each species treated with PEC at 65 percent creosote in treating solution. Both faces of the cross-section stakes are shown.

TABLE 8. *– Number of stakes per set of 10 at each treatment by retention by species combination with percentage cross section penetrated with creosote.*

Preservative	Creosote	Cross section that was penetrated (%)			
		0 to 25	26 to 50	51 to 75	76 to 100
	%	(no of stakes)			
Douglas-fir					
PEC	65			3	7
	30			1	9
	15		3	4	3
	7.5		3	5	2
P1/P13	65			4	6
	30			5	5
	15			5	5
	7.5		4	6	
Red maple					
PEC	65		1	2	7
	30		1	5	4
	15				
	7.5				
Red oak					
PEC	65				10
	30			7	3
	15		3	7	
	7.5		8	2	
Pl/P13	65			1	9
	30			7	3
	15		8	2	
	7.5	1	6	3	
Southern pine					
PEC	65				10
	30		1	5	4
	15		3	4	3
	7.5		1	6	3
P1/P13	65				9
	30			3	7
	15			5	5
	7.5			4	6

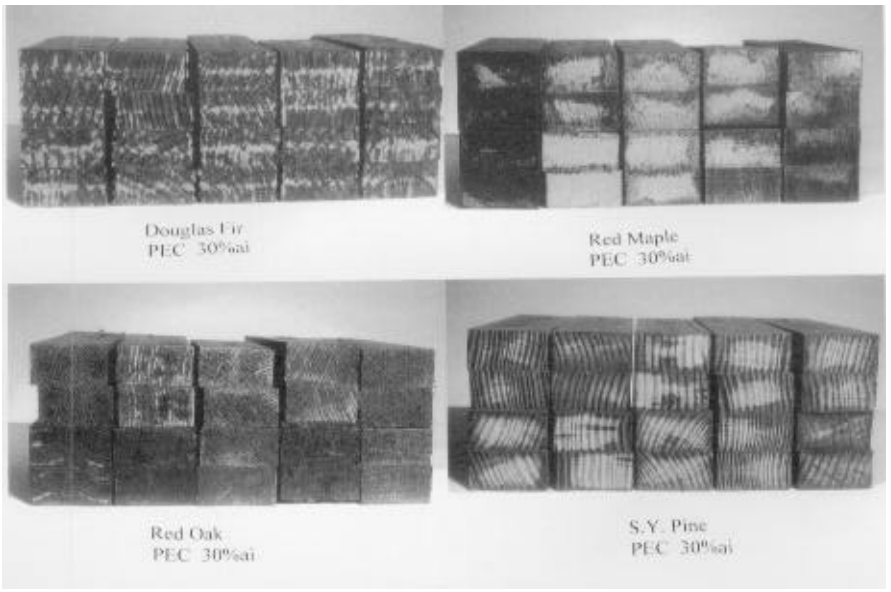

Figure 3. – Cross section of 10 stakes of each species treated with PEC at 30 percent creosote in treating solution. Both faces of the cross-section stakes are shown.

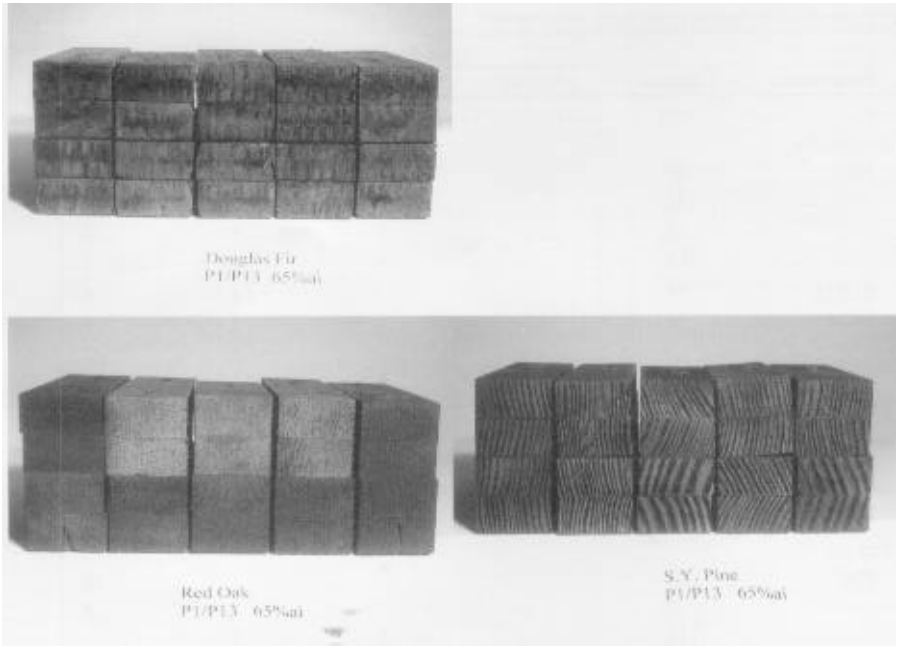

Figure 4. – Cross section of 10 stakes of each species treated with P1/P13 at 65 percent creosote in treating solution. Both faces of the cross-section stakes are shown.

domly selected from the original set of 30) per treatment. The PEC-treated stakes were analyzed at both locations. Stakes treated with the reference P1/P13 creosote were analyzed only at FPL. Both laboratories were in general agreement in their analysis of PEC-treated stakes, except that retention determined at FPL tended to be slightly greater than those determined at CSIRO in Douglas-fir, red maple, and southern pine but not in red oak. At the highest creosote concentration of PEC-30W, the retention determined by chemical analysis was equivalent to that determined by weight gain. At the three lower concentrations of creosote in the PEC-30W treating solution, retention determined at both laboratories for PEC-30W was only about half the retention determined on the basis of weight gain.

With the reference creosote (P1/P13) in Douglas-fir and southern pine **(Table 7),** there was general agreement between retention levels determined by weight gain and those by extraction analysis, especially at the higher concentrations. In red oak at all concentrations, retention determined by extraction analysis was much lower than the retention determined by weight gain.

The greatest cross-sectional area and uniformity of treatment with both formulations occurred at the highest creosote concentrations **(Tables 8** and **9; Figs. 2** through **5).** The percentage cross-sectional area penetrated by creosote in stakes treated with P1/P13 at 7.5 percent through 30 percent creosote was not strongly influenced by creosote concentration. However, the tendency for "zebra or coon-tail patterns" (that is, treatment of only the latewood within annual rings) to occur, increased as creosote concentrations decreased **(Figs. 2** through **5).**

DISCUSSION AND CONCLUSIONS

Treatment of four U.S. wood species with PEC W30 was generally comparable to results of treatment with the reference creosote P1/P13 except that slightly greater variability in creosote loading occurred with PEC. The uniformity of cross-sectional area penetrated by either formulation was dependent on creosote concentration. Whether or not the difference between gravimetric and analytical methods used to determine creosote retention in wood treated with the lowest three concentrations of PEC was a true result or reflected a procedural difficulty with solvent extraction of creosote from PEC-treated wood was not resolved. However, it has always been difficult to solvent extract creosote from PEC-treated timber. This is believed to be associated with the pigment particle 'blocking" mechanism. This hypothesis is now being investigated.

The results of this study indicate that U.S. species of wood can be treated with both creosote formulations. Additional work on processing is required to demonstrate the potential for U.S. species of wood to meet Australian and U.S. standards with both formulations. Work is ongoing to investigate the surface chem-

TABLE 9. – Creosote retention [a] in outer 6 mm and in central core treated stakes.

Species	Creosote concentration	Treatment	Extraction weight percent creosote		
			Inner	Outer	Whole
	(%)		(kg/m^3) (pcf)	(kg/m^3) (pcf)	(kg/m^3) (pcf)
Douglas-fir	65	PEC	340 (21)	399 (25)	415 (26)
		P1/P13	344 (21)	275 (17)	341 (21)
Southern pine	65	PEC	406 (25)	383 (24)	447 (28)
		Pl/P13	383 (24)	341 (21)	421 (26)
Red oak	65	PEC	159 (10)	157 (9)	90 (6)
		P1/P13	199 (12)	240 (15)	233 (14)
Red maple	65	PEC	289 (18)	341 (21)	338 (21)
		P1/P13			

[a] Average of five stakes. Samples taken at midlength of stakes

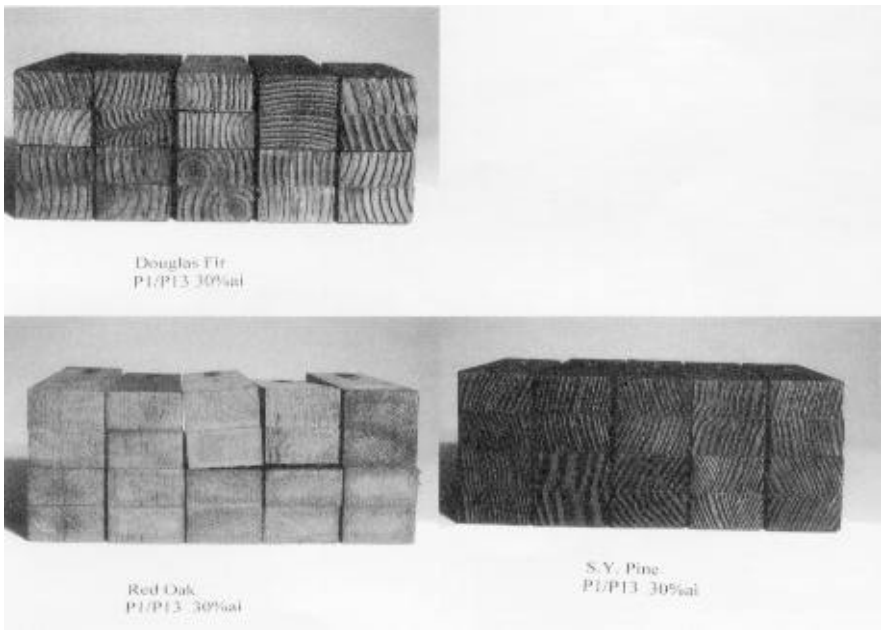

Figure 5. – Cross section of 10 stakes of each species treated with P1/IP13 at 30 percent creosote in treating solution. Both faces of the cross-section stakes are shown.

istry (16) and interfacial phenomena to the pigment particle interactions in the emulsion (13), especially within the micro-structure of the treated commodity, during and after pressure treatment, and especially related to the surface drying mechanism and the difficulty encountered in extracting the creosote during soxhlet extraction.

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