# TREATMENT OF PEELER CORES WITH WATER-DISPERSIBLE PRESERVATIVE FORMULATIONS[1]

## H. M. Barnes

Professor
Mississippi Forest Products Utilization Laboratory
P.O. Drawer FP, Mississippi State, MS 39762

## ABSTRACT

Hardwood peeler cores were conditioned and treated with either water-dispersible pentachlorophenol (PWD) or water-dispersible zinc naphthenate (ZWD). The results indicated that adequate treatment could be obtained with either system using conventional treating cycles and methods of conditioning stock for treatment. Treatment was related to heartwood content and was better for the ZWD system. This difference was attributed to the swelling ability of the ZWD system. Sweetgum treated better than yellow-poplar. Presteaming dry stock led to improved treatment and flatter preservative gradients.

*Keywords:* Preservatives, pressure treatment, kiln-drying, steam-conditioning, steaming, yellow-poplar, sweetgum, pentachlorophenol, zinc naphthenate.

## INTRODUCTION

Water-dispersible preservative formulations have become increasingly important in recent years concurrent with the energy and environmental concerns of the wood-preserving industry. The reformulation of oil-borne systems into water-dispersible systems based on emulsion/dispersion technology has continued. Initial work was done with pentachlorophenol to form either emulsifiable systems (Hatcher 1980, 1981; Barnes 1983; Kirchner and Taylor 1983) or water-miscible/soluble systems (Amundsen et al. 1978; Hudson 1983, 1984). These systems have greatly reduced oil content and produce commodities with clean, paintable surfaces. An additional advantage of these reformulations has been the reduction of oil-containing process effluent streams.

During this time period, a plethora of aqueous systems based on biocides previously carried in oil solvents have been developed. Notable among these biocides are copper-8-quinolinolate, salts of the naphthenic acids, 3-iodo-2-propynyl butyl carbamate, benzothiazoles, and others (Behr 1979, 1981; Mathur 1983; Nicholas 1983; Barnes 1985).

Although hardwoods will become increasingly important as a feedstock for the forest-based industries, they offer unique problems from a treatment mechanics viewpoint. Aside from fence posts and crossties, little information is available on the efficacy of waterborne treatments for hardwoods. With CCA treatment, failure due to soft rot attack has been well documented (Levy 1978). While an excellent review of the preservative treatment of hardwoods is available (Thompson and
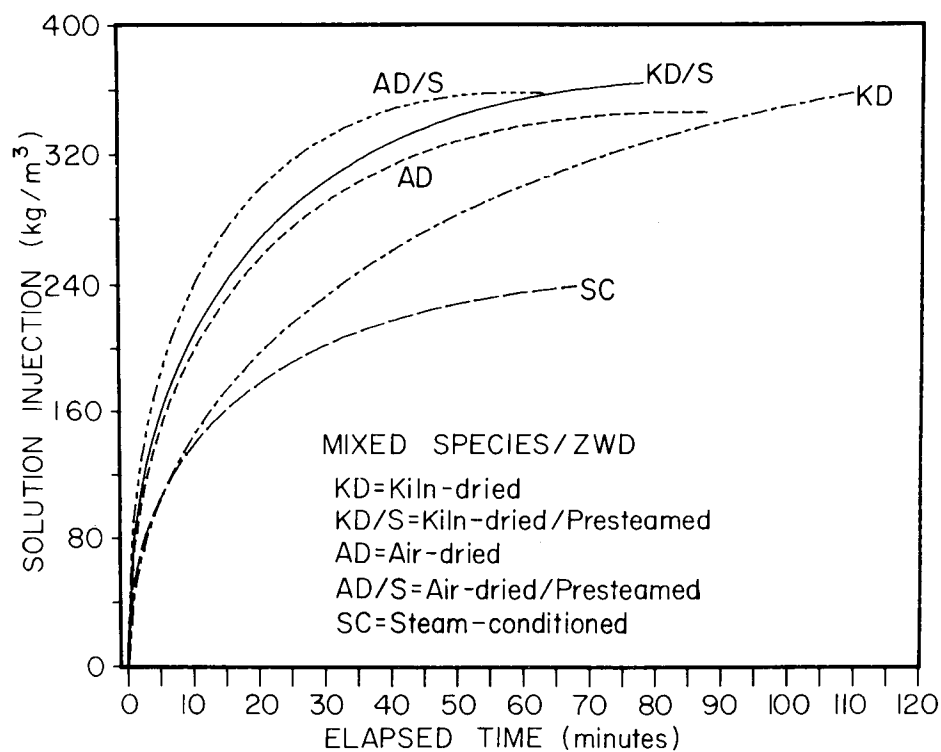
Fig. 1.   Fitted injection curves for full-cell treatment of hardwood and pine veneer cores with water-dispersible zinc naphthenate (ZWD).

TABLE 1.   *Effect of conditioning method on treatment of peeler cores of mixed species with two water-dispersible preservative formulations.*

| Conditioning method | Gross injection, kg/m³ | Time to refusal, min | Relative contribution to gross injection, %[a] | | |
|---|---|---|---|---|---|
| | | | Pine | Sweetgum | Yellow-poplar |
| Water-dispersible Zinc Naphthenate (0.2% as zinc) | | | | | |
| Kiln-dried | 357 | 110 | 32 | 50 | 18 |
| Kiln-dried/presteamed | 366 | 78 | 63 | 21 | 16 |
| Steam-conditioned | 238 | 68 | 42 | 39 | 18 |
| Air-dried | 355 | 88 | 37 | 41 | 21 |
| Air-dried/presteamed | 363 | 63 | 43 | 38 | 19 |
| Average | 336 | 81 | 43 | 38 | 19 |
| Water-dispersible Pentachlorophenol (5.0% chlorinated phenols) | | | | | |
| Kiln-dried | 202 | 60 | 66 | 21 | 13 |
| Kiln-dried/presteamed | 262 | 93 | 35 | 56 | 9 |
| Steam-conditioned | 267 | 68 | 42 | 31 | 27 |
| Air-dried | 258 | 83 | 43 | 33 | 24 |
| Air-dried/presteamed | 322 | 70 | 29 | 52 | 19 |
| Average | 262 | 75 | 43 | 39 | 18 |

[a] Based on the weight gain by species as a percentage of the total weight gain for a given charge.
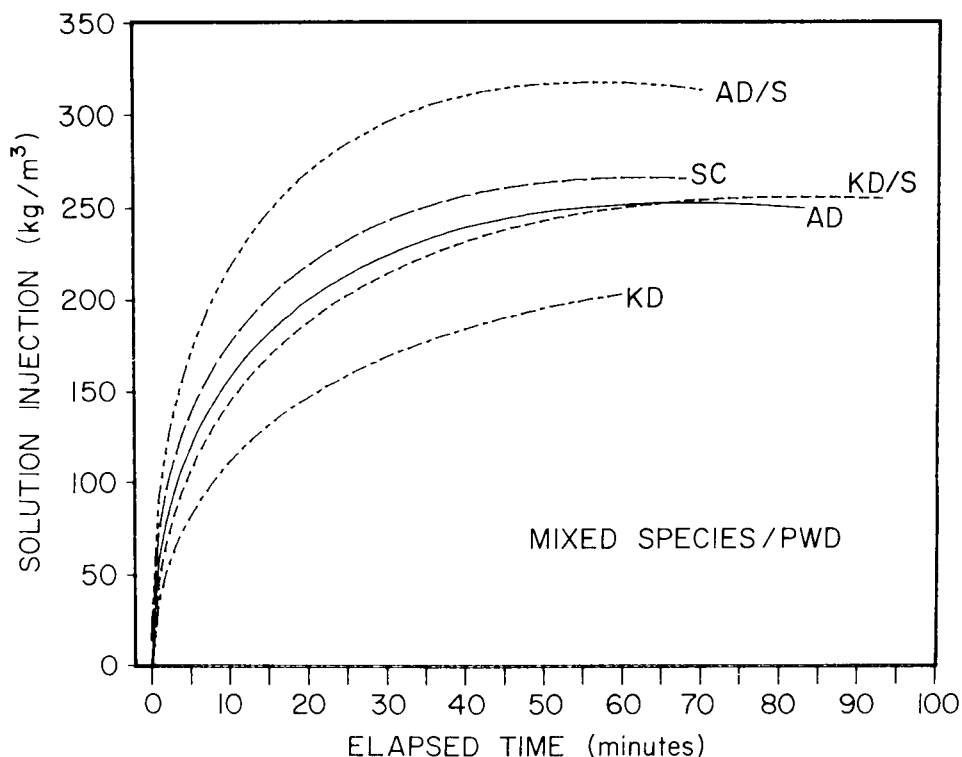
FIG. 2.   Fitted injection curves for full-cell treatment of hardwood and pine veneer cores with water-dispersible pentachlorophenol (PWD) [Letter codes same as Fig. 1].

Koch 1981), no data are available on treatment with new waterborne preservative systems.

The purpose of this research was to develop a data base on treatment of hardwoods with water-dispersible preservative formulations. Data on southern pine are included for comparison.

## EXPERIMENTAL PROCEDURES

### Design

A series of three studies was conducted. In the first, peeler cores were treated in mixed-species charges using the full-cell process. Two charges for each conditioning method were treated. Each charge consisted of equal volumes of each of the species described below. The conditioning method prior to treatment was varied in order to evaluate the effect of various methods on treatment. In a second study, slabbed hardwood cores were treated using full-cell or Lowry process in order to assess treatment process and species differences. In the final investigation, kiln-dried quarter-round hardwood fencing boards were presteamed and treated using both treating processes.

### Materials

Green sweetgum (*Liquidambar styraciflua* L.), yellow-poplar (*Liriodendron tulipifera* L.), and southern pine (*Pinus* sp.) plywood peeler cores, nominally 127
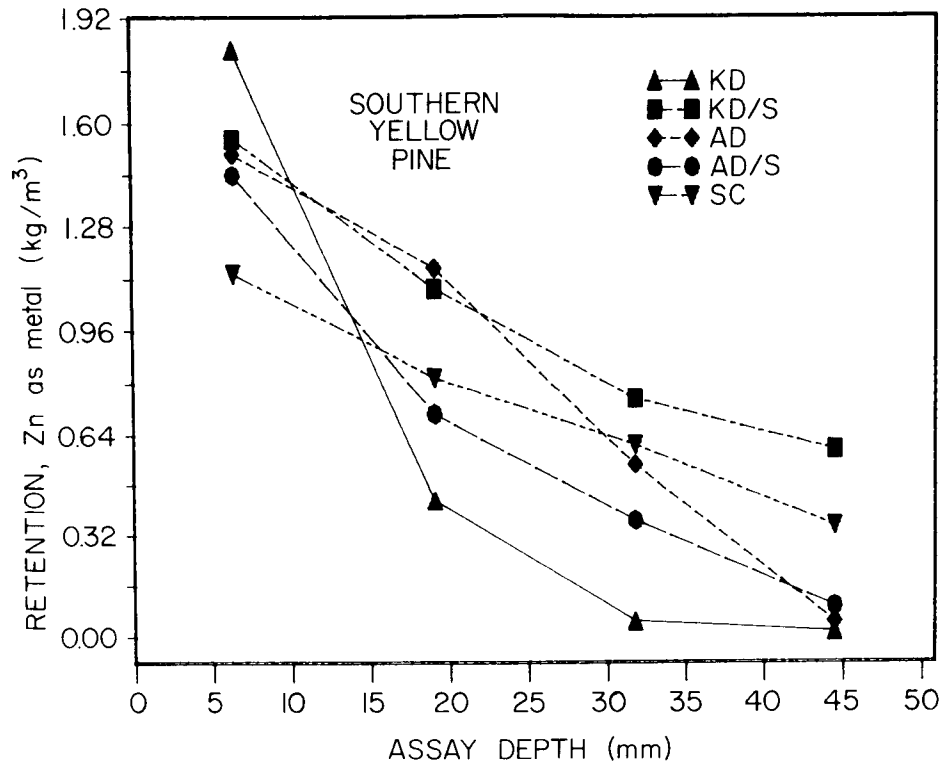
FIG. 3.  Zinc gradients by conditioning process for southern yellow pine treated with the ZWD system [Letter codes same as Fig. 1].

mm in diameter, were obtained from a cooperator. Heartwood content of hard-wood cores was determined by inspection and averaged 51% and 85% for gum and poplar, respectively. All cores were 2.59 m in length as received. Pine cores averaged 35% heartwood as determined using benzidine dihydrochloride/sodium nitrate heartwood indicator (AWPA 1983). Some of the cores were slabbed on two sides to 89 mm prior to further processing. The slabs were retained and treated as quarter-round fencing boards as described later.

### Conditioning

Cores and slabs were either kiln-dried (KD), steam-conditioned (SC), or air-dried (AD) prior to treatment. Air-drying was accomplished by crib-stacking cores on an air-drying rack, covering to exclude rainfall, and drying for a minimum of three months to a moisture content of 25% or less prior to treatment.

The kiln schedule used was identical to that described by Kelso (1974). Maximum dry-bulb temperature was 77 C and fan speed was 2.54 mps. Target moisture content was 15% or less. A presteaming (S) cycle was used to precondition some of the dried stock. The cycle consisted of raising the temperature to 116 C over a 30-minute period, direct steaming at 116 C for one hour, and applying vacuum of −88 kPa for 30 minutes.

A six-hour steam conditioning cycle reduced the green material such that the
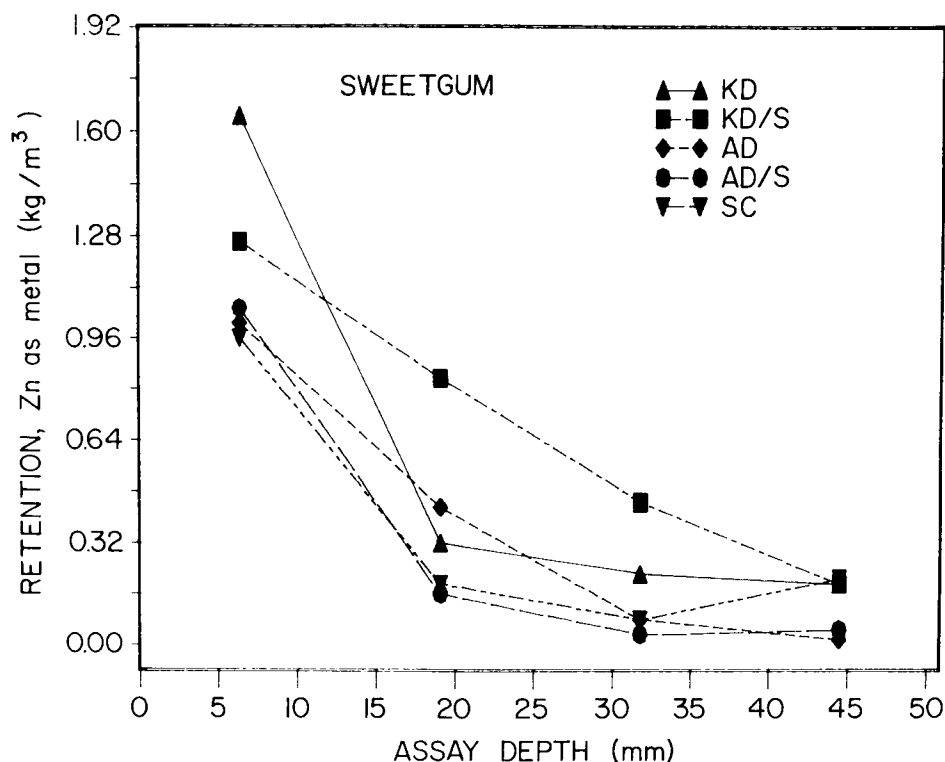
Fig. 4.   Zinc gradients by conditioning process for sweetgum treated with the ZWD system [Letter codes same as Fig. 1].

moisture content to below 40%. The cycle consisted of raising the temperature to 116 C in 30 minutes, direct steaming at 116 C for 4.5 hours, and applying a final vacuum of −88 kPa for one hour.

### Pressure processes

Cores and slabs were treated using either a full-cell or Lowry empty-cell process. Stock was weighed and measured to determine volume prior to loading into the cylinder. For a typical full-cell charge, samples were loaded into the treating retort, which was evacuated (−88 kPa) for one hour and then filled with preservative while under vacuum. Pressure was then increased at the rate of 34.5 kPa/min to a maximum pressure of 1,034 kPa and maintained until refusal was reached. The cylinder was vented to atmospheric pressure, and the excess preservative was removed. Samples were removed, and the net retention was determined by weight gain.

The Lowry process differed from the full-cell process in that no initial vacuum was used. The cylinder was filled with preservative at atmospheric pressure via a pump, and the pressure was increased to 1,034 kPa as before. The pressure was held constant until refusal was reached. A 30-minute final vacuum (−88 kPa) completed the process.

In both processes pressure was applied through a scale tank containing preser-
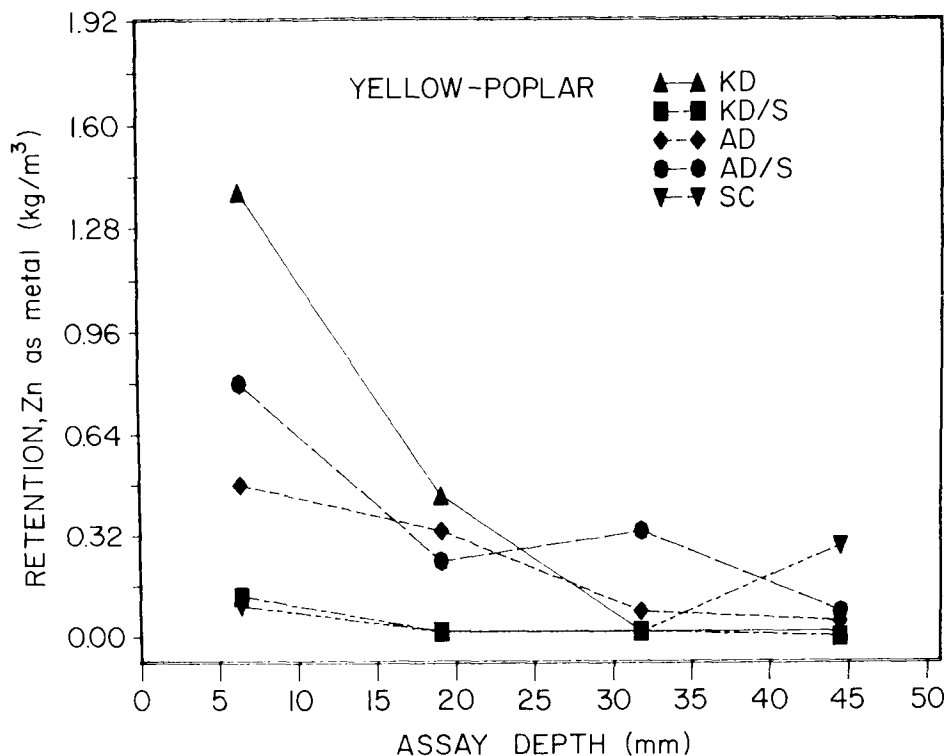
Fig. 5.   Zinc gradients by conditioning process for yellow poplar treated with the ZWD system [Letter codes same as Fig. 1].

vative so that the gross injection curve could be measured. Injection curves were fitted to an equation of the form: gross injection (kg/m$^3$) = A + Bt + C (sqrt(t)) where A, B, and C are constants and t is time in minutes.

### Preservative systems

Two new-generation preservative systems were chosen for investigation. The first was a proprietary water-dispersible penta system (PWD) and was supplied as a 25% concentrate of total chlorinated phenols. The treating emulsion was prepared by diluting concentrate with water to form a 5% solution. The second system was a commercial water-dispersible zinc naphthenate concentrate (ZWD) and was supplied as an 8% zinc (as metal) concentrate. A treating emulsion of 0.2 or 1.0% (Zn as metal) was prepared by dilution with water.

Emulsions were stored in separate working tanks and agitated. Fresh emulsion was added every 3–5 charges as needed. All effluent streams, except the vacuum condensate, were recycled into the working tanks.

### Analysis

Preservative gradients were determined from borings taken from the treated stock. Slabbed peeler cores were bored through the rounded face. Slabs were bored parallel to the wide face of the slab. Borings from like species and from charges
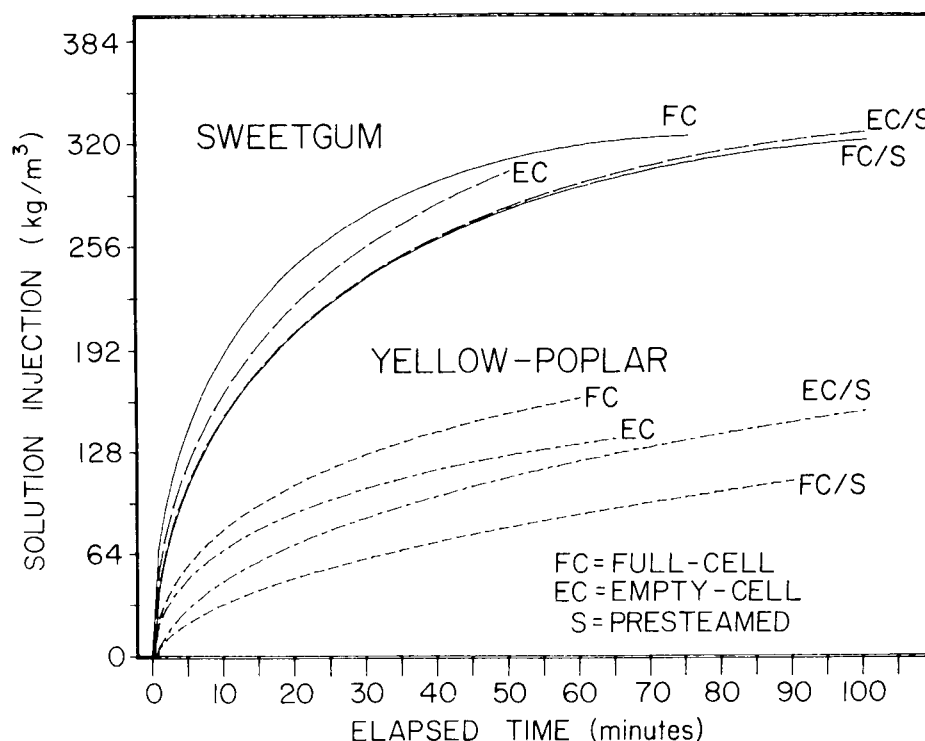
Fig. 6.   Comparison of gross injection curves for kiln-dried and presteamed hardwoods treated with the ZWD system using full- and empty-cell processes.

receiving the same conditioning method and pressure process were segmented into assay zones and combined for analysis.

*a. Assay for pentachlorophenol.* —PWD treating emulsions were assayed by high performance liquid chromatography (HPLC) using the method described by Barnes (1983). Extraction was with either methylene chloride or chloroform. Treated wood borings were ground to 10 mesh on a Wiley mill, sonicated in methanol for 30 minutes, and allowed to stand overnight. The filtered extract was analyzed using HPLC.

*b. Assay for zinc.* —ZWD emulsions (0.1 g aliquots) were wet-ashed in boiling, concentrated nitric acid, diluted appropriately with water, and analyzed using a Perkin-Elmer Model 305 atomic absorption spectrometer that was fitted with a zinc cathode ray tube using an air/acetylene mixture. Wood borings were ashed in Vycor Crucibles in a muffle furnace at 600 C. The ashed residue was dissolved in hot, concentrated nitric acid, diluted with water, and analyzed as described by Barnes (1983).

### RESULTS AND DISCUSSION

#### Effect of conditioning method

It is difficult to analyze gross injection curves in mixed species charges and obtain a valid picture of treatment since each species treats differently. For commercial charges, however, it would be desirable to treat mixed charges since

TABLE 2.    *Effect of treating process on the treatment of hardwood peeler cores treated with two water-dispersible preservative formulations.*

| Volume treated, m³ | % Heartwood | Process | Injection, kg/m³ | | Time to refusal, min. | Injection[a] ratio, % |
|---|---|---|---|---|---|---|
| | | | Refusal | Net | | |
| *Water-dispersible Zinc Naphthenate (1.0% as zinc)* | | | | | | |
| Sweetgum/KD | | | | | | |
| 0.149 | 51.9 | Full-cell | 325 | — | 75 | — |
| 0.134 | 49.1 | Empty-cell | 301 | 288 | 50 | 95.7 |
| Sweetgum/KD-Presteamed | | | | | | |
| 0.075 | 39.6 | Full-cell | 322 | — | 100 | — |
| 0.076 | 39.6 | Empty-cell | 326 | 320 | 100 | 98.0 |
| Yellow-poplar/KD | | | | | | |
| 0.151 | 88.3 | Full-cell | 162 | — | 60 | — |
| 0.153 | 81.3 | Empty-cell | 136 | 102 | 65 | 75.3 |
| Yellow-poplar/KD-Presteamed | | | | | | |
| 0.076 | 94.7 | Full-cell | 156 | — | 90 | — |
| 0.074 | 94.7 | Empty-cell | 152 | 152 | 100 | 100.0 |
| *Water-dispersible Pentachlorophenol (5.0% as chlorinated phenols)* | | | | | | |
| Sweetgum/KD | | | | | | |
| 0.154 | 57.8 | Full-cell | 296 | — | 160 | — |
| 0.154 | 57.8 | Empty-cell | 293 | 218 | 100 | 74.3 |
| Sweetgum/KD-Presteamed | | | | | | |
| 0.154 | 72.2 | Full-cell | 325 | — | 80 | — |
| 0.152 | 70.0 | Empty-cell | 355 | 341 | 130 | 96.4 |
| Yellow-poplar/KD | | | | | | |
| 0.152 | 84.3 | Full-cell | 306 | — | 160 | — |
| 0.152 | 84.3 | Empty-cell | 181 | 160 | 135 | 88.5 |
| Yellow-poplar/KD-Presteamed | | | | | | |
| 0.152 | 73.7 | Full-cell | 266 | — | 130 | — |
| 0.152 | 91.5 | Empty-cell | 203 | 203 | 130 | 100.0 |

[a] Ratio of net injection/refusal injection.

production costs would be lowered. To obtain an estimate of the relative contribution of each species to the total injection, the ratio of the weight increase for individual samples to the total weight increase in a given charge was calculated. Summing these percentages by species yielded the total contribution of each species to gross injection (Table 1). Pine had the highest gross absorption for all conditioning methods, followed by sweetgum and yellow-poplar.

*a. Gross injection curves.*—A summary of the fitted full-cell injection curves by conditioning method is shown in Figs. 1 and 2 for the ZWD and PWD systems, respectively. Coefficients of determination ($R^2$) ranged from 82.8% and 99.4% and averaged 96.5% and 92.1% for the ZWD and PWD systems, respectively. Air-dried material yielded the highest rate of injection, followed by kiln-dried and steam-conditioned material (Fig. 1) treated with the ZWD system. A similar trend was seen for the PWD-treated material except that steam-conditioned material treated more rapidly than air-dried or kiln-dried material (Fig. 2). Presteaming improved the injection rates of dried stock with both systems.
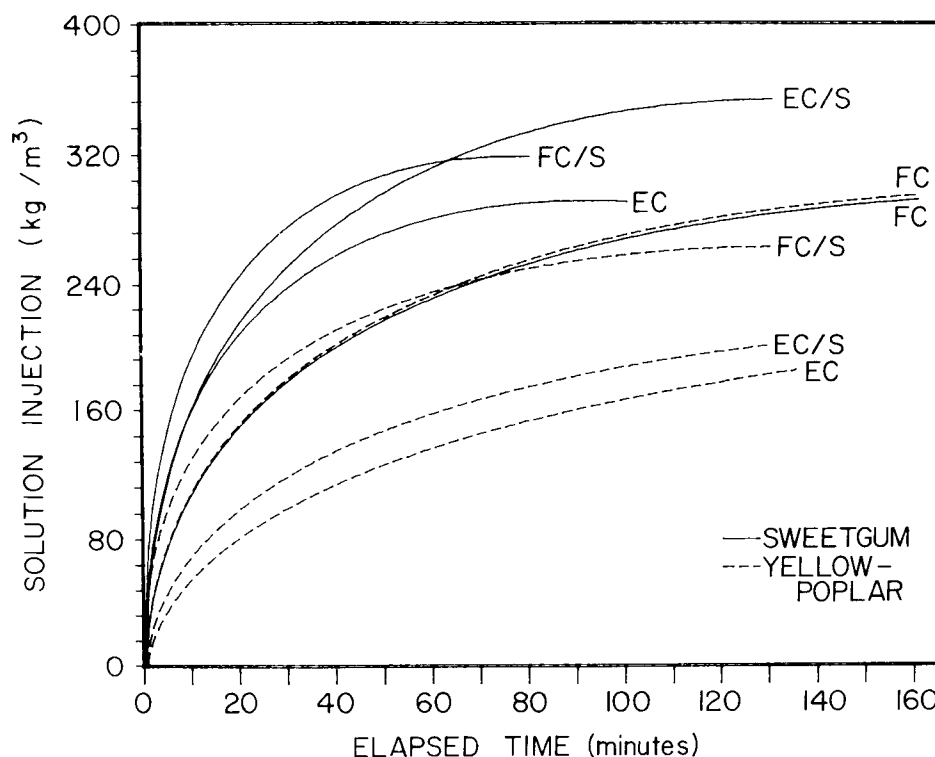
FIG. 7. Comparison of gross injection curves for kiln-dried and presteamed hardwoods treated with the PWD system using full- and empty-cell processes [Letter codes same as Fig. 6].

Presteaming improves injection rate presumably because it reduces pit aspiration in pine while also hydrolyzing the pit membranes and encrusting materials in both pine and hardwoods (Nicholas and Siau 1973). This is especially true for kiln-dried pine where pit aspiration and setting of extractives could be more of a problem.

With the ZWD system, air-dried and steam-conditioned stock treated to refusal in about the same time, with steam-conditioned stock reaching a much lower gross injection. Gross injection for all other conditioning methods was identical. Kiln-dried stock reached approximately the same final injection as did air-dried stock, but the time-to-refusal was much greater (Table 1).

Results with the PWD system were much more varied. Presteamed/air-dried stock yielded the greatest gross injection, while kiln-dried material yielded the lowest gross injection. Gross injection for the other conditioning methods was about the same (Table 1).

The rate for steam-conditioned stock was lowest for the ZWD system and intermediate for the PWD system. No consistent trend was shown with the PWD system. These results can be attributed to differences in the preservative formulations. Gross injections for the ZWD system were significantly higher than those for the PWD system with the exception of steam-conditioned material. Across all conditioning methods, the ZWD system had an average gross injection of 336 kg/m$^3$ compared to an injection of 262 kg/m$^3$ for the PWD system. The relation-
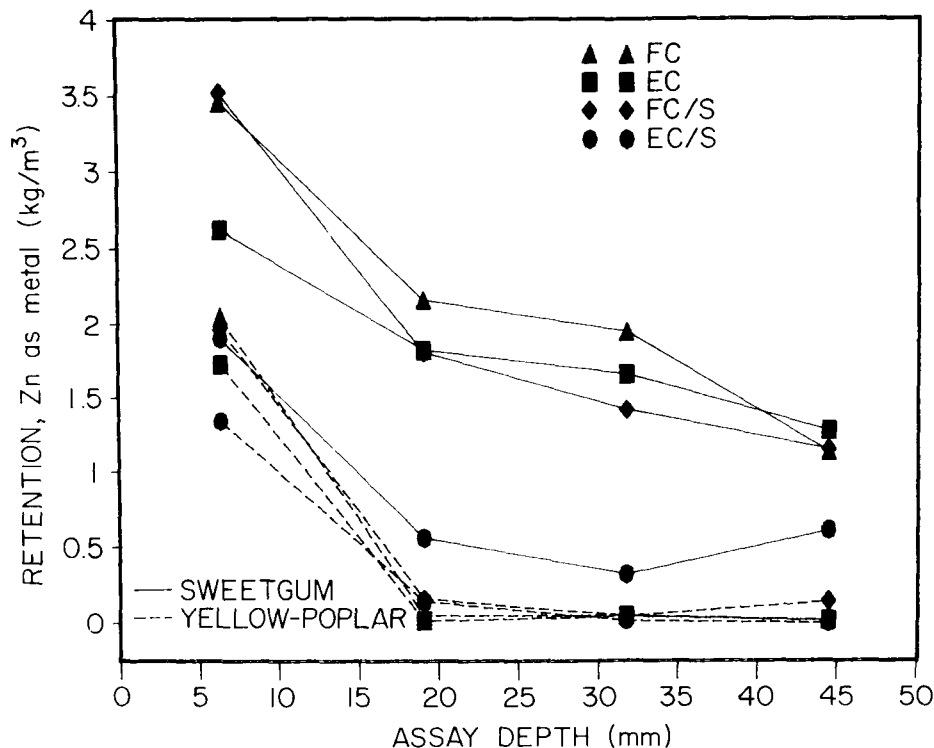
FIG. 8.    Effect of process on zinc gradients obtained with kiln-dried and presteamed hardwoods treated with the ZWD system [Letter codes same as Fig. 6].

ship is similar to that found between traditional waterborne systems, which swell the wood structure and preservatives carried in oil which are nonswelling solvents. Other factors may also be responsible for the observed differences. First, the ZWD system may be a better swelling agent because of its lower active-ingredient strength and solvent content. The water-dispersible concentrate was formed by dissolving zinc naphthenate in a solvent, then adding wetting agents and surfactants. The PWD concentrate was made by dissolving penta in a P-9 Type A (AWPA 1983) solvent amended with cosolvents to improve penta solubility. The addition of surfactants and wetting agents allowed for water dispersion. Also, it may be that the micelles formed during the emulsifying of the active ingredients were smaller in the ZWD system.

b. *Preservative gradients.* — In this initial study, preservative gradients were measured only in the stock treated with the ZWD system. Previous work (Barnes 1983) has shown that acceptable gradients through the sapwood zone can be obtained for material treated with 5% PWD to a 224 $kg/m^3$ gross. Gross injection with the PWD system averaged 262 $kg/m^3$ across all conditioning methods. Zinc gradients by conditioning method are shown in Figs. 3–5 for pine, sweetgum, and yellow-poplar, respectively. For all conditioning methods, pine gradients were higher than those for sweetgum, and yellow-poplar yielded the lowest gradients. This trend is related to the heartwood content of each species and the higher
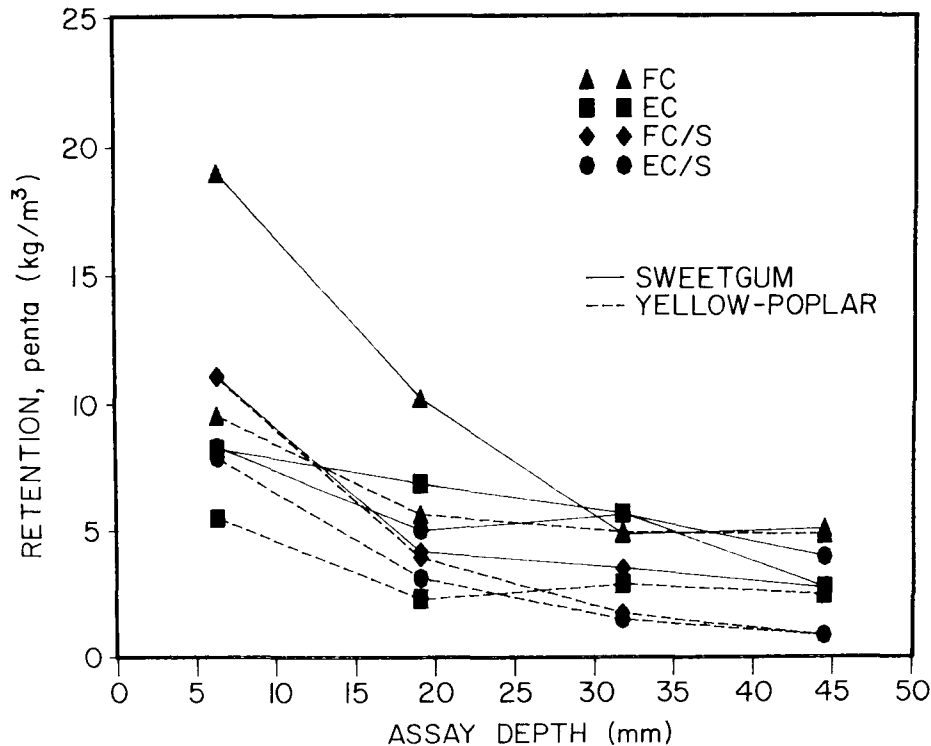
FIG. 9.  Effect of process on penta gradients obtained with kiln-dried and presteamed hardwoods treated with the PWD system [Letter codes same as Fig. 6].

treatability of pine as compared to hardwoods. Across all conditioning methods, pine averaged 35%, while sweetgum and yellow-poplar averaged 51 and 85%, respectively. In each instance, sapwood was completely penetrated. Heavy loading of preservative in the outer assay zone was noted for kiln-dried material of each species. Gradients were much steeper for this stock. Presteaming tended to flatten the gradients for dried stock. In addition to reducing pit aspiration and hydrolyzing pit membranes and encrusting materials as indicated previously, presteaming would also relieve stresses induced during drying, which would lead to a better, more uniform treatment. Stress relaxation using high temperature and humidity is a common practice in the kiln-drying of wood. With respect to conditioning method, analysis of the gradients showed the following general trend across all species: presteamed > dried > steam-conditioned. This is due partly to the higher moisture content of the steam-conditioned stock. Steam-conditioned stock averaged 32% moisture content across all species, while dried stock averaged 24%.

The results suggest that steaming greatly improves the treatment of dried stock regardless of the preservative system used. Across species, pine was more easily treated than hardwoods, with gum being more easily treated than poplar. The fact that sapwood penetration was complete in all species indicates that hardwood and pine cores can be successfully treated in mixed species charges.
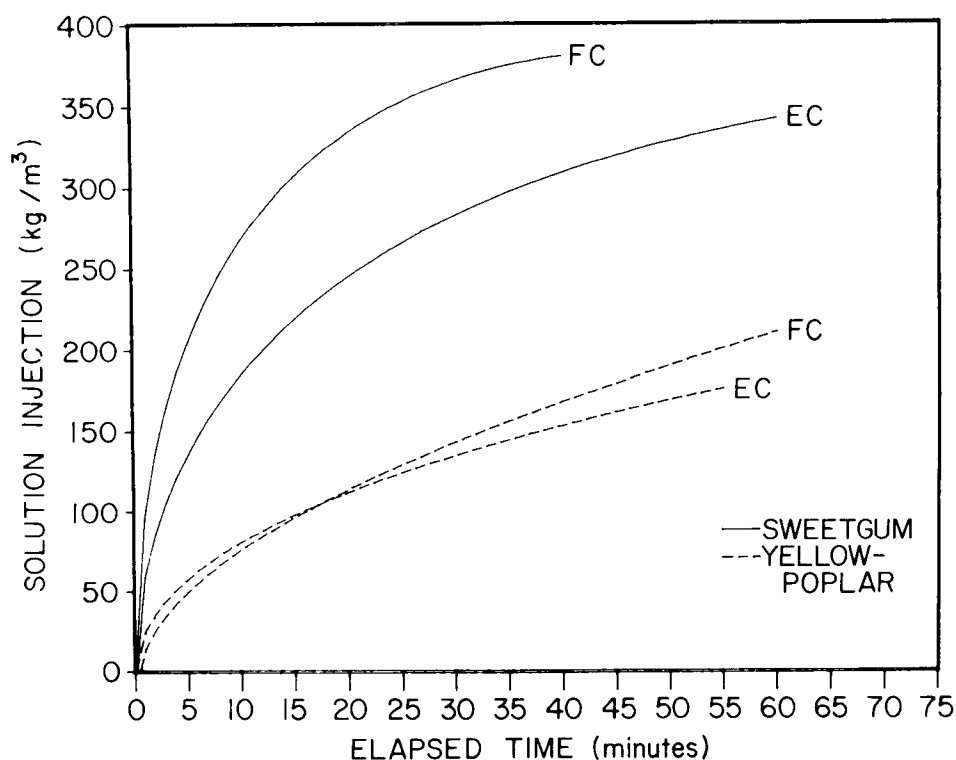
FIG. 10. Injection curves for fencing boards treated with the ZWD system [Letter codes same as Fig. 6].

## Effect of process

In order to isolate the effect of the process on the treatment of hardwoods, a series of kiln-dried and presteamed/kiln-dried slabbed cores were treated with both preservative systems.

*a. Gross injection curves.* —Gross injection curves for kiln-dried and kiln-dried/ presteamed gum and poplar treated with the ZWD system using empty- and full-cell processes indicate an excellent fit of the injection curves, with the lowest $R^2$ being 95.8% (Fig. 6). The injection curve for full-cell treatment was only slightly above the empty-cell curve for kiln-dried stock of both species. Excellent fits were obtained for the combined data for each species. A summary of the injection data is given in Table 2. The gum accepted a much higher gross injection than the poplar (Fig. 6), again indicating the relatively small sapwood zone in poplar cores and the difficulty in treating poplar heartwood.

Presteaming improved the preservative injection for all combinations of species and process except the yellow-poplar that was treated by the full-cell process. This is similar to the trend found in the conditioning study. Steaming tends to relieve drying stresses, remove surface set, and swell the wood. All of these actions tend to improve the movement of preservative into the wood and suggest that steaming dried hardwoods should be a pretreatment.

Comparable data for empty-cell and full-cell treatment of hardwood cores with
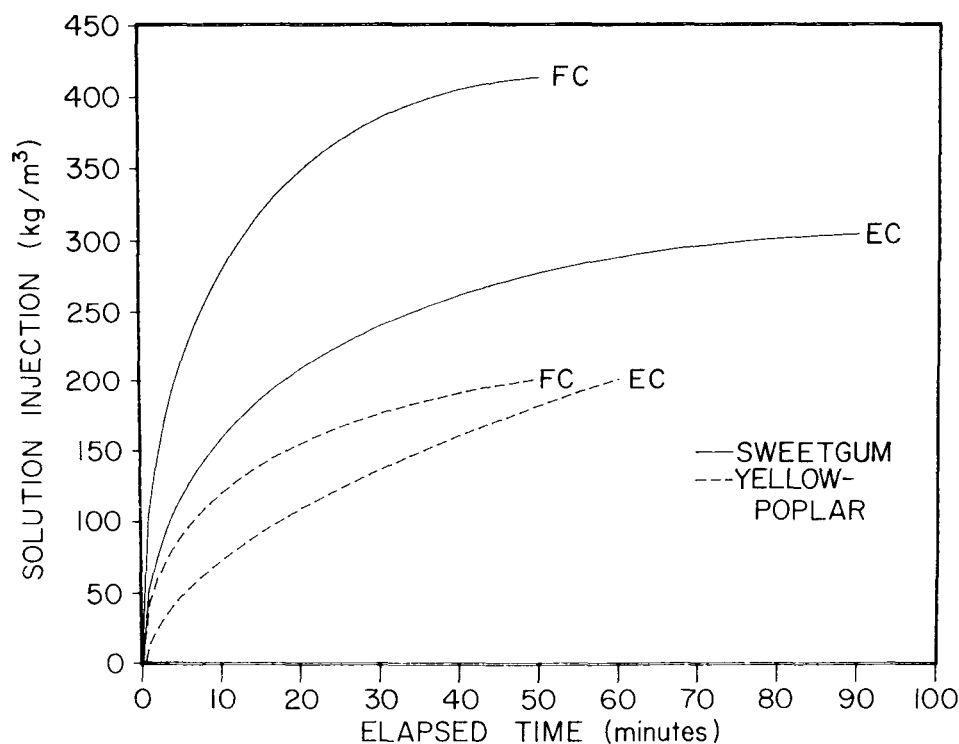
FIG. 11.   Injection curves for fencing boards treated with the PWD system [Letter codes sames as Fig. 6].

the PWD system are shown in Fig. 7 and Table 2. Injection times were much longer than those for the ZWD system. Gum accepted a higher gross retention than poplar. Empty-cell treatment of gum and full-cell treatment of poplar yielded similar injection curves. For both species, full-cell injection was faster than the

TABLE 3.   *Summary of the charge data for treatment of hardwood and pine quarter-round fencing boards with two water-dispersible preservative formulations.*[a]

| Species | Process | Volume treated, m³ | Retention, kg/m³ | | Time to refusal, min | Injection[b] ratio, % |
|---|---|---|---|---|---|---|
| | | | Refusal | Net | | |
| Water-dispersible Zinc Naphthenate (1.0% as zinc) | | | | | | |
| Sweetgum | Full-cell | 0.099 | 444.8 | — | 40 | — |
| | Empty-cell | 0.099 | 340.8 | 305.6 | 60 | 89.7 |
| Yellow-poplar | Full-cell | 0.106 | 232.0 | — | 55 | — |
| | Empty-cell | 0.107 | 208.0 | 208.0 | 60 | 100.0 |
| Water-dispersible Pentachlorophenol (5.0% as chlorinated phenols) | | | | | | |
| Sweetgum | Full-cell | 0.101 | 462.4 | — | 50 | — |
| | Empty-cell | 0.104 | 328.0 | 328.0 | 60 | 100.0 |
| Yellow-poplar | Full-cell | 0.106 | 182.4 | — | 50 | — |
| | Empty-cell | 0.103 | 200.0 | 200.0 | 60 | 100.0 |

[a] All stock was kiln-dried, then presteamed for one hour prior to treatment.
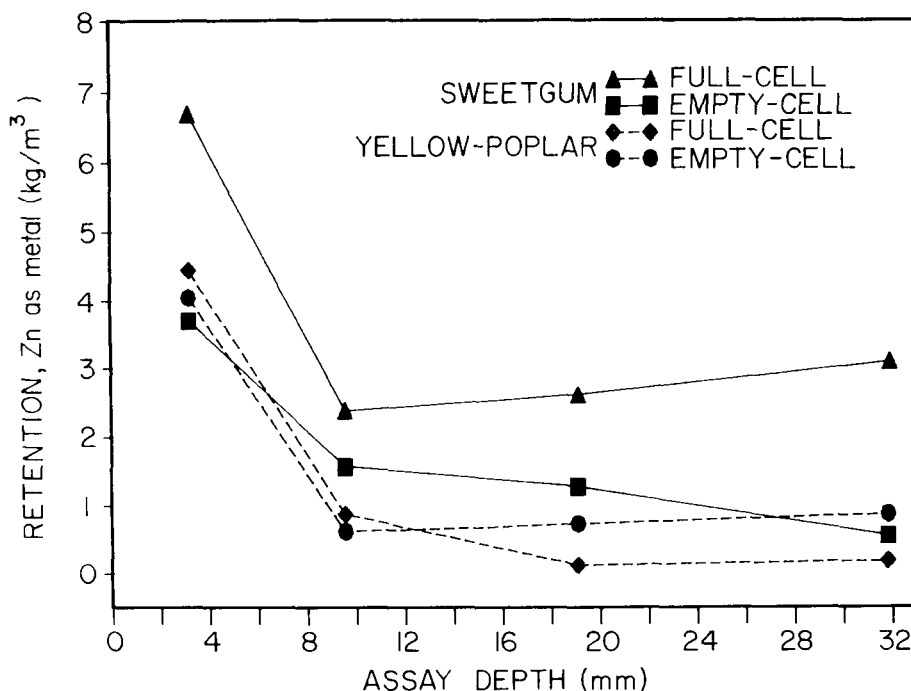[b] Ratio of net injection/refusal injection.

FIG. 12.   Zinc gradients for fencing boards treated with the ZWD system.

empty-cell rate. Presteaming of sweetgum yielded higher gross and net injections for empty-cell treatment and an equivalent net injection for full-cell treatment in half the time required for kiln-dried stock. Presteaming also reduced the time required for full-cell treatment of yellow-poplar.

Comparisons of gross and net retentions between preservative formulations indicate no consistent trends. Comparable net injections with sweetgum were found with both systems for full-cell treatments. The reverse was true for yellow-poplar, with the PWD treatment yielding much higher net injections. Empty-cell treatment of gum with the ZWD system yielded a higher net than treatment with the PWD system while the reverse was true for empty-cell treatment of yellow-poplar.

*b. Preservative gradients.*—Gradients by species and process are shown in Fig. 8 for the ZWD system and Fig. 9 for the PWD system. The relative magnitude of the gradients was less important than their shape. The zinc gradients for sweetgum were less steep than those for yellow-poplar. Gradient shape for both species was similar across processes with the full-cell gradient being slightly higher than the gradient for empty-cell treatment. The steep gradient for poplar was indicative of narrow sapwood zone for this species.

Inspection of borings taken from PWD-treated stock indicated a highly variegated penetration that varied widely across individual samples depending on heartwood content and checking pattern. This variegation was also evident when the gradients were analyzed (Fig. 9). The same general trend found with the ZWD system was evident with PWD treatment. No heartwood penetration was evident
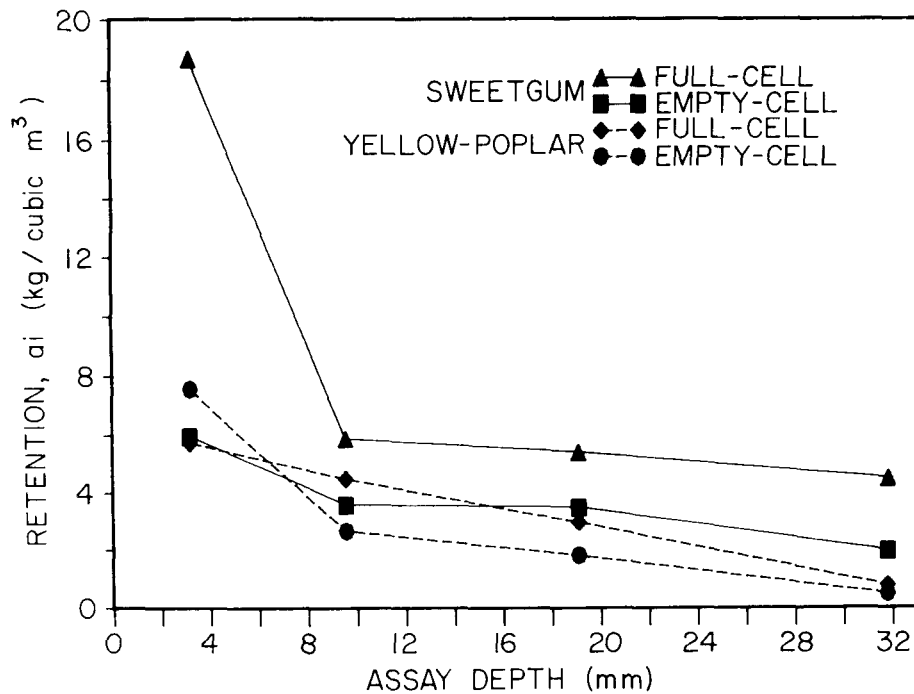
Fig. 13.   Zinc gradients for fencing boards treated with the PWD system.

with the PWD system and gradients for the PWD system tended to be steeper. This may reflect the difference in the swelling ability of these systems.

Presteaming improved both gradient shape and preservative penetration (Figs. 8, 9) with the greatest improvement for the empty-cell treatment. Presteaming tended to equalize full-cell and empty-cell gradients for both preservative systems although the effect was greatest for the PWD system. This difference was consistent with a larger micelle size, differences in solvent system, and lower swelling ability of the PWD system.

### Treatment of quarter-round fencing boards

Injection curves and charge data for the treatment of presteamed fencing boards (Figs. 10, 11; Table 3) reflect the much higher sapwood content of this stock. In general, adequate penetration and retention of preservative were noted, regardless of process, species, or preservative system. Across species, full-cell injection rate was equivalent to or greater than the empty-cell injection rate. The injection rate in gum was higher than that for poplar.

Higher refusal injection levels were found for the ZWD system although treatment times across species and processes were equivalent for both systems. These data indicate adequate treatment of hardwood stock containing high percentages of sapwood.

Preservative gradients for fencing boards (Figs. 12, 13) for the ZWD and PWD systems indicate good treatment. These gradients are much flatter than those obtained previously, reflecting the high sapwood content in this stock.

## SUMMARY AND CONCLUSIONS

The data presented indicate that complete treatment of hardwood sapwood for above-ground exposure can be obtained with water-dispersible zinc naphthenate and pentachlorophenol systems using conventional treating cycles and conditioning methods. Results varied, but better treatment was generally obtained with the water-dispersible zinc naphthenate system than with the water-dispersible pentachlorophenol system. This improved treatment was attributed to the difference in the swelling ability of the systems. Injection times tended to be shorter with the ZWD system.

Treatment quality tended to be related to the heartwood content of the species with species or products containing less heartwood tending to treat more easily. Sweetgum generally treated more easily than yellow-poplar and yielded higher retentions with gradients that were less steep with both systems. Adequate empty-cell treatment was obtainable, but full-cell treatment tended to be faster and yielded flatter, more uniform gradients. Presteaming dried stock improved both the injection rate and the preservative gradients while producing better penetration and flatter gradients.

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