DURA-TREET* II, A WATER DISPERSIBLE PENTACHLOROPHENOL

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INTRODUCTION

During the past several decades, penta petroleum wood treating has become increasingly popular. Pentachlorophenol (penta) an effective chemical against wood organisms causing decay and rot is safe to handle, and clean and easy to use. In terms of preservative cost, it has been and remains by far the least expensive of the three major preservatives. However, when the cost of petroleum carrier is added, the penta petroleum process has become as costly as other processes. There appears to be no end to the increase in petroleum costs and there may even be substantially reduced availability in the future. All of these factors indicate a need for treating processes which will keep the pressure treating industry competitive and profitable in the face of intense competition from other materials.

Over 12 years ago, Idacon initiated an ongoing research program looking for a clean treating process and methods for reducing the cost of applying pentachlorophenol in pressure treating processes. In 1978, at the AWPA meeting in Washington, D.C., Idacon announced a water-borne pentachlorophenol process which completely eliminated the need for oil. This process is the subject of U.S. patent number 4,090,000, Canadian patent number 1,066,002, British patent number 1,538,691, as well as patents in France, Brazil and continuing applications in New Zealand, Australia and several other countries. This process, called Dura-Treet I, was passed over for commercialization in favor of the one that is the subject of this paper, as Dura-Treet II appears to fit the immediate needs of the pressure treating industry better than Dura-Treet I.

Dura-Treet II represents a less radical departure from established practice than its predecessor. Dura-Treet II is pentachlorophenol dissolved in a mixture of hydrocarbon and co-solvents meeting the distillation requirements of P9 Oil Type A, and greatly exceeds the pentachlorophenol solvency requirements of P9 Type A. A concentrate of pentachlorophenol in this special P9 solvent is dispersed in water. This dispersion appears to be submicron, although actual size determinations remain to be made.

This dispersion of oil-borne penta in water is stable for months, penetrates southern pine readily and seems to penetrate Douglas-fir just as well as penta petroleum. The result is a clean treatment that reduces usage of hydrocarbon by approximately 85 percent when compared with a 7 percent penta petroleum solution.

From the laboratory tests and commercial usage, there appears to be little, if any, difference in leaching rates, efficacy against fungi, point of deposition of pentachlorophenol in the cell, corrosion rates, strength, or other factors to differentiate it from penta petroleum 7 percent.

*Dura-Treet is a Trademark of Idacon, Inc.

PROCESS DESCRIPTION

As previously stated, Dura-Treet II does not represent a substantial departure from methods or equipment presently used to treat with penta petroleum. No special equipment or chemicals are needed. The Dura-Treet II, dispersible pentachlorophenol system, can be introduced into any plant currently treating with penta petroleum. There is, therefore, need for extremely minor or no additional capital investment or change in established treating practices.

Standard practice in using this new process is for a 40 percent concentrate to be delivered which is diluted with a dispersing agent prior to mixing further with water. In order to prepare the solution concentration for actual use, normally around 5 percent to 7 percent, the concentrate is metered into a predetermined quantity of water in the work tank. Experience has shown that violent agitation or high sheer equipment is not required for mixing. If the concentrate is pumped directly into the top of the work tank, the dispersion mixes well during simple recirculation of the solution. There has been no problem with "fallout". However, in general practice it is recommended that the concentrate be injected into the water recirculation line, or that an agitator be installed to avoid any possibility of undispersed concentrate settling on the bottom of the tank, which might occur if large quantities were injected rapidly. It has been found that the injection point should be near the center of the tank, since dispersion is much more difficult if the material contacts the walls of the vessel.

Experimentation and use have shown that the makeup water need not be preconditioned or treated. Available water from the plant may be used. In most cases it is possible to use effluent water from the steam conditioning of green material and in some cases, pond water. While pond water has been used commercially, it is not recommended as high concentrations of calcium, magnesium and other materials present in some pond waters could cause instability of the dispersion. Experience has shown that a small percentage will drop out as concentrate over several months if there is complete absence of agitation or circulation. This concentrate does not redisperse with agitation alone. It is necessary to add fresh concentrate containing dispersant before the deposited material will redisperse. However, in the cases where this has happened, the deposited material has been successfully redispersed and used for treating.

Once the dispersion is at the proper concentration, treating proceeds as normal with only minor adjustments in time and pressure. Since Dura-Treet II is a clean treat no final steaming is necessary and very little or no plant contamination need be experienced from preservative drip.

The process can be used either on dry or steam conditioned stock. It is also applicable to either the full-cell or empty-cell process (1). However, the preferred usage is empty-cell, since the weight increase due to the treatment is no more than with penta petroleum. There is no need to kiln dry after treating in order to reduce weight for shipping. Experience has shown that a rapid weight loss is experienced during the first few days exposure of the treated material to the atmosphere. This results in a lighter finished product compared to penta petroleum, and represents a substantial saving in shipping costs compared to shipping wood that has been treated by a full-cell process.

One of the questions that is often encountered with this process is whether the kickback is compatible with the dispersion of the work tank. There is complete compatibility, as the fixation in the wood does not require a chemical reaction or cause a kickback that reacts with treating solution. The only effect of kickback, or condensate from steam conditioning is to reduce the solution concentration. As previously stated the condensate from steam conditioning has been used for solution makeup water with no adverse effects.

COMPARATIVE EVALUATION

The previous review of Idacon's new process indicated many of the economic advantages. However, the main thrust of the AWPA is directed very strongly at protecting the user of treated wood and not at industrial economics. Only the technical aspects of whether the product meets rigid standards of quality are considered. Consequently, during development of Dura-Treet II Idacon's work was directed towards a series of comparative tests to determine whether this new product met the rigid standards and demands of the wood treating industry. First, however, laboratory treating tests were necessary, as comparative tests are lengthy and expensive, so that only the most worthy projects go to final comparative tests.

After Idacon had found that it was possible to produce a stable dispersion of penta in P9 oil with water a series of treating tests were undertaken in a pilot plant consisting of three cylinders capable of performing the normal functions of pressure treating. The smallest of these cylinders was a mere 4 in. x 18 in. and is normally used for initial screening of products or processes to eliminate the product which doesn't penetrate or which shows undesirable characteristics. The second cylinder (6 in. x 52 in.) is used primarily for treating standard 18 x 0.75 x 0.75 in. stakes for stake tests, and for screening tests with larger test pieces.

When a product survives screening in the smaller cylinders, the 10 in. x 12 ft. pilot cylinder is used to treat charges of lumber of various sizes, fence posts and pole stubs to determine whether the product has desirable characteristics for commercialization. Obviously, Dura-Treet II survived this screening, although the screening was not viewed optimistically in the beginning. This was due to the past history of penta in P9 oil mixing with water to form emulsions. All treaters have had experience with this, and the penetration, to put it mildly, is somewhat less than desirable. In contrast, penetration with Dura-Treet II proved to be excellent. In fact, it appears to be slightly superior to penta petroleum.

After the dispersible penta survived the preliminary treating evaluation, testing was undertaken to determine whether this product met the necessary standard. For this purpose, it was compared with an accepted standard, namely 7 percent pentachlorophenol in P9 oil, Type A. These tests compare resistance to leaching with water, threshold retention by soil block tests, electron microscope examination, metal corrosion comparisons, and strength comparisons. All of the above were conducted on southern yellow pine sapwood. Other wood species remain to be studied.

SOIL-BLOCK TESTS

Procedure

Three-quarter inch (0.75 x 0.75 x 0.75 in.) cubes of southern pine sapwood treated by Idacon with pentachlorophenol in standard and experimental carrier systems were conditioned to constant weight in an environmental chamber and tested using the standard soil-block procedure (ASTM Standard D 1413-76). Five blocks for each retention group were exposed to cultures of each of two fungi, *Gloeophyllum trabeum* and *Poria monticola*, in 8-ounce decay chambers. In addition, a sixth block was placed in a sterile chamber for weight control purposes. Untreated southern pine blocks were used as controls in the study.

RESULTS

(a) Soil block tests

There was no apparent difference in efficacy of treatment between the standard and experimental carrier systems (2). The lowest retentions that prevented decay for each combination of carrier and test organism were as follows

	Gloeophyllum trabeum	*Poria monticola*
Standard PCP (lb./ft.^{3})	0.330	0.330
Experimental PCP (lb./ft.^{3})	0.345	0.345

For some unexplained reason, blocks treated with dispersant and water showed some effectiveness in preventing decay. The average weight loss was 7.1 percent. The comparable value for blocks treated with P9 oil was 45.5 percent while blocks treated with water only, showed a 57.1 percent weight loss. The above tests were conducted at Mississippi State University by Dr. Warren Thompson.

(b) Leaching tests

With a water based preservative system, it is necessary to confirm that the preservative is fixed in the wood and that there is no problem with leaching of preservative. The comparative leachability of Dura-Treet II versus penta petroleum was therefore examined. Data are expressed as a percentage of the original pentachlorophenol retention and percentage loss of total pentachlorophenol after leaching. This test was conducted on 0.75 x 0.75 x 0.75 in. blocks of southern yellow pine sapwood, treated in accordance with AWPA Standard M10-77. These blocks were then subjected to leaching in accordance with AWPA Standard M11-66. After leaching, the relative permanence was determined assaying the blocks and aliquots of the leach water by lime ignition (AWPA Standard A5-79).

When first attempts were made to check leachability, the Standard method (AWPA M11-66) seemed a tedious method fraught with hazards. Due to the low leachability of penta and inability to analyze blocks until after the leaching, an attempt was made to develop a new leach test. Commercially treated posts were sawed to produce sawdust, which was then analyzed, by lime ignition, divided into aliquots, leached and analyzed again. This alternative method was completely unacceptable. Duplication was impossible and results were ridiculous. In contrast,the method described in M11-66 worked well, giving accurate and consistent results, even though it was time consuming.

The extremely small amount of preservative which was leached by the AWPA standard M11-66 test was below the limits of the normal lime ignition method (AWPA Standard A5-79) test capabilities. Consequently, an aliquot had to be concentrated for the pentachlorophenol determination. There were some variations in individual blocks due to wood density difference and other factors. Blocks treated with four different retentions of pentachlorophenol in toluene and Dura-Treet II were leached. Both systems display the same leaching curve after approximately 72 hours. With high retentions, the pentachlorophenol in toluene appeared to initially leach more readily. In contrast the opposite effect is observed with low retentions. It should be recognized however, that such a small amount of pentachlorophenol is leached from blocks treated with either system that such conclusions are of questionable value. The test indicated a leaching rate of the same order of magnitude for Dura-Treet II and penta petroleum. The rate at which both leach is extremely low, but more importantly, the tests showed that the dispersion of oil-borne penta in water displayed no greater leachability than the penta petroleum system (3).

(c) Electron microscope investigation

With the use of a scanning electron microscope it is possible to determine whether the preservative (pentachlorophenol) is placed in the same location within the wood structure, when wood is treated with either penta petroleum or dispersible treatments. This is an extremely important comparison since improper location of preservative could cast doubt on its efficacy. Comparisons of element maps for chlorine distribution produced from the dispersible treatment and standard penta petroleum at depths of 0-1 and 3-4 cm showed no differences in the distribution of preservative. Quantitative analysis for chlorine at specimen depths of 0-1 and 3-4 cm showed a chlorine peak height corresponding to 0.73 and 0.47 lb./ft.^{3} of pentachlorophenol.

The X-ray investigations were interpreted by Dr. Warren Thompson of Mississippi State University as showing no difference between the standard and experimental solvent systems insofar as location of chlorine in the cell wall is concerned (4).

(d) Corrosion tests

With memories of a previous pentachlorophenol preservative system (2% penta-creosote) that proved extremely corrosive to plant equipment, Idacon submitted the process to Southwestern Laboratories for testing according to NACE Standard TM-91-69 (1972 Revision) Laboratory Corrosion Testing of Metals for the Process Industries. The metal specimen used for the test was 1020 carbon steel. The specimens were subjected to a 5% penta petroleum and a dispersion of 5% oil-borne penta in water. The results of the corrosion rate for three 48-hours test periods are:

Test Solution	1st Period	2nd Period	3rd Period	Average
5% penta petroleum	1.40 mils/yr.	1.27 mils/yr.	1.15 mils/yr.	1.27 mils/yr.
5% Dura-Treet II	0.60 mils/yr.	0.62 mils/yr.	0.61 mils/yr.	0.61 mils/yr.

The test shows Dura-Treet II to be less corrosive than standard penta petroleum. While a laboratory test cannot be used to guarantee the corrosion rate because field and laboratory conditions will never be exactly the same, a test of this type is, however, a good indication of the relative corrosive properties of each solution (5).

COMMERCIAL EXPERIENCE AND USAGE

Once the comparative evaluations were complete, the question still remained: Will it work in full scale, day to day commercial treating? To answer this question a full scale test was started in late March, 1979.

The first full scale plant run, using Dura-Treet II was a charge of posts and lumber. This charge was steam conditioned and consisted of 431 cubic feet of fence posts ranging from two and one-half to seven inches in diameter. Six cubic feet of rough lumber was included for test purposes. A dispersed treating solution of 3.9 percent pentachlorophenol (as measured by lime ignition) was prepared. Since this was going to be a test, and the run temporary in nature, the standard empty cell process (1) was modified. Instead of initial air, the cylinder was filled by vacuum, since this procedure was standard practice in this plant.

After the cylinder was full, pressure was applied and increased to 155 psi and maintained for one hour and fifteen minutes. After treatment, a final vacuum was applied for thirty-five minutes. Work tank gauges indicated a net retention of 0.615 pcf. Increment borings were taken from a large percentage of the posts for analysis.

Results of the analysis ranged from 0.29 pcf to 1.7 pcf with the majority of results falling in the 0.55 to 0.75 pcf range. Borings from a seven inch post were analyzed in one-half inch increments. The results were as follows: 0.0 to 0.5 in. 0.76 lb./ft.^{3}; 0.5 to 1.0 in. 0.50lb./ft.^{3}; 1.0 to 1.5 in. 0.44 lb./ft.^{3}; and 1.5 to 2.0 in. 0.40 lb./ft.^{3}.

Additional treatments also proved successful. These in-plant tests were discontinued at this point and the dispersion stored for a period of time in the plant, after which a permanent change to this new process was approved and instituted. Since these early tests, other plants have converted to Dura-Treet II. As of February, 1980 approximately 400,000 fence posts are in use and an estimated 20,000 cubic feet per week are being treated, with increased usage beyond this forecasted for the immediate future.

The fence posts from these plants go primarily to areas of high biological activity, i.e., the Gulf Coast. Continued monitoring of fence posts in service is planned.

Small quantities of lumber have also been treated. No problems were encountered, except that the clean treatment does not have the same appearance or color as the accustomed product. Therefore, attempts to incorporate dyes into the dispersion were made, which proved to be successful. Most colors are available, but the most economical of those that penetrate into the wood with the dispersion are brown, green and red.

There has been no commercial usage of Dura-Treet II on poles as yet. However, extensive testing was undertaken on Class 3 and Class 4 poles 25 ft. in length. These were treated in an extremely well designed and instrumented experimental facility. The test cylinder was 3 ft. x 26 ft. and was accompanied by all the auxiliaries normally found in the best of treating plants.

The purpose of the test was to evaluate the effectiveness of Idacon's dispersible penta as a preservative treatment for both dry and green southern pine poles by determining penetration and retention in charges treated under normal conditions. Thirty-two southern pine poles (which were a mixture of class 3's and class 4's) were treated in the study. Sixteen of these poles were green and sixteen were dry. The poles were arranged into eight groups of four poles. Each group made up a charge, and consisted of all green or all seasoned poles. Dispersible penta concentrate was diluted with water to a 7 percent concentration in one of the work tanks. The target retention was 0.6 lb./ft.^{3} (by gauge) or 0.45 lb./ft.^{3} (by assay). The four charges of dry poles were treated first, followed by the four charges of green poles. Prior to loading the charges for treatment borings of the sapwood portions were removed from each pole. After each charge, the treating solution was analyzed for its penta content.

Following treatment, each charge was assayed by taking a boring sample consisting of five borings taken at the groundline of each of the treated poles. These borings were divided into the following separate assay zones: 0.0 - 0.5 in., 0.5 - 2.0 in., and 2.0 - 2.5 in. (where no heartwood was encountered).

When all treatments were completed, a penetration boring was taken from each pole (6).

It was found, (Table 1) that the process ensures proper penetration of the PCP, reacts normally to treating variables and offers no barrier to commercial usage on poles. Observations from the tests were that

1. Pressures over 100 psi are necessary, and
2. Using 30 minutes final vacuum after treating eliminated the foam normally observed on the poles.

Table 1. — SUMMARY OF RESULTS

Charge No.	Cubic Ft. in Charge	Seasoning* Condition	Solution Strength %	Actual Gross (Gals.)	Actual Net (Gals.)	Retention by Gauge (lb/ft.^{3})	Retention by Assay (lb./ft.^{3}) 0.0-0.5″	0.5-2.0″	2.0-2.5″	Treating Cycle Steam (h.)	Vacuum (h.)	Pressure** (lbs.)	Final Vacuum (h.)
1	41.47	Dry	6.75	71	35	0.49	0.48	0.35	—	2	1	103	1/4
1R	41.47	Dry	6.51	19	8	0.60	0.77	0.62	0.54	0	0	107	1/4
2	40.29	Dry	6.51	103	44	0.62	0.52	0.34	0.27	0	0	107	1/4
3	43.36	Dry	6.44	86	32	0.40	0.54	0.36	0.27	0	0	105	1/2
4	40.91	Dry	6.50	82	43	0.58	0.62	0.38	0.32	0	0	105	1/2
5	45.66	Green	6.53	57	28	0.35	0.91	0.40	0.37	5	1	105	1/2
6	44.14	Green	6.32	57	35	0.42	0.86	0.28	0.21	6	1	105	1/2
7	43.01	Green	7***	69	23	0.33	1.08	0.58	0.23	6	1	200	1/2
8	43.18	Green	6.74	72	27	0.36	1.14	0.63	0.38	6	1	200	1/2
2R	40.29	Dry	6.71	50	18	0.87	0.86	0.53	0.40	0	0	105	1/2
6R	44.14	Green	6.50	50	37	0.89	1.47	l0.41	0.27	0	0	200	1/2

*Average Sapwood Moisture Content of Dry Poles - 28.6%
Average Sapwood Moisture Content of Green Poles - 39.5%
** Initial Air Pressure was 20 Pounds For All Treatments
*** Solution was Strengthened to a Calculated 7% Before Charge No. 7

CONTINUING EVALUATION

Several sets of standard 18 x 0.75 x 0.75 in. stakes have been treated with with the experimental treatment and 7 percent penta petroleum to varying retentions as required by ASTM Standard D-1758 (AWPA Standard M7-73). These stakes were placed in selected test plots they will be monitored and results reported, as they become available. It is also planned in the future to commercially treat a number of carloads of poles which will then be placed in service.

Continuing commercial production will be monitored and the results of products in commercial usage carefully observed and reported.

CONCLUSIONS

The laboratory tests have indicated the Dura-Treet II process to be equal to a penta petroleum treatment when evaluated for fungal resistance, resistance to leaching and placement of preservative (pentachlorophenol) in the cell walls. It has also been shown that corrosion problems are not inherent to the system. Preliminary results of strength or static bending tests have proceeded far enough to show that there are no adverse effects when Dura-Treet II is compared to penta petroleum.

Plant scale tests on posts and lumber plus commercial usage have shown Dura-Treet II to be a viable and efficient process which lends itself to commercial plant conversion. Large scale experimental tests also indicate the process to be suitable for use on poles.

These results, plus the economics of a water carrier with potential energy savings, indicates a system which deserves consideration from those interested in maintaining a strong, competitive wood preserving industry.

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