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Evaluation of a Boron-Nitrogen, Phosphate-Free Fire-Retardant Treatment. Part III. Evaluation of Full-Size 2 by 4 Lumber per ASTM Standard D 5664-95 Method C*

ABSTRACT: The purpose of this work was to evaluate the effects of a new boron-nitrogen, phosphate-free fire-rerardant (FR) formulation on the initial strength of No. 1 southern pine 2 by 4 lumber and its potential for in-service thermal degradation. The lumber was evaluated according to Method C of the D 5664 standard test method. The results indicated that for lumber exposed at 150°F (66°C) for 108 days, FR treatment and redrying significantly (a £ 0.10) decreased initial bending strength by about 13% compared to that of untreated controls. No significant difference occurred in the rate of strength loss over time of exposure. This infers that, after accounting for the initial reduction in strength, the field performance of FR-treated lumber should be comparable to that of untreated lumber. From a practical standpoint, the effect of FR treatment on maximum load capacity was similar to that on bending strength. Treatment significantly reduced work to a maximum load by 299, but it had no differential effect on the rate of change in this property when the lumber was exposed to elevated temperatures. Although modulus of elasticity was not significantly changed by treatment, this property was significantly increased by extended exposure at high temperature. In summary, the reduction in mechanical properties for FR-treated 2 by 4 lumber occurred at a rate no different than that for matched untreated lumber when exposed to elevated temperature.

KEY WORDS: fire-retardant, treatment, thermal degradation, high temperature, lumber

This is the third report of a three-part evaluation of the effects on strength and strength retention at high temperature for a new boron-nitrogen, phosphate-free fire-retardant (FR) treatment. Part I of this research program [1] evaluated Douglas-fir plywood as specified in ASTM D 5516-96 [2]. Part II [3] evaluated small clear specimens of Douglas-fir, white spruce, and southern pine as specified in Methods A and B of ASTM D 5664-95 [4].

The work reported here was performed according to Method C of ASTM Standard D 5664-95. This study was conducted for three rasons. First, this is the first time that anyone haas reported in the open literature on the procedures described in ASTM D 5664-95 in the version that was finally approved by the American Society for Testing and Materials (ASTM). The D 5664 standard evolved, after several iterations, from nonstandardized test methods originally and independently developed by code bodies or industrial associations as building product evaluation criteria. While the final ASTM standard is similar to the original evaluation methods used to qualify current commercially available FR formulations, it is not identical. Second, these data and the experience we gained through this work may be used to develop an understanding of the attainable precision and bias of ASTM D 5664-95 or to improve its design or economy. Third, until the recent introduction of this new FR, no phosphate-free FR formulation was available in the commercial market. Our research may have direct benefits to consumers with respect to safety and long-term serviceability if this new phosphate-free FR is eventually accepted by national building codes and standardized through the American Wood Preservers' Association (AWPA).

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Objective

The objective of this study was to evaluate the effect of a new boron-nitrogen, phosphate-free FR formulation on the mechanical properties of full-size, nominal 2- by 4-in. (standard 38- by 89-mm) lumber (hereafter called 2 by 4) exposed to elevated temperature (150°F (66°C)) and 75% RH for up to 108 days We also wanted to gain an understanding of the precision and bias of ASTM D 5664-95.

Method and Materials

The experimental design consisted of two treatments (FR-treated and untreated) and two environmental exposures (no exposure and exposure for 108 days at 150°F (66°C) and 75% RH). Each experimental group consisted of 50 specimens. Results of destructive tests on full-size southern pine 2 by 4s were analyzed to determine the effect of treatment on several mechanical properties and the potential for thermal-induced loss in properties when exposed to elevated in-service temperatures.

Material

Southern pine No. 1 & better, 12-ft (3.7-m)-long 2 by 4 lumber (208 pieces), was obtained from a mill in southwest Arkansas.

Each piece was evaluated for modulus of elasticity (MOE) using transverse vibration. Pieces with the four highest or four lowest MOE values were culled. The remaining 200 pieces were then sorted into four MOE-matched groups of 50 pieces each. This technique ensured that each group had nearly identical distributions of high. middle, and low MOE specimens. Such a technique helps ensure that any post-experimental difference in properties can be attributed directly to the experimental variables and not to chance variation.

The grade-dictating defect for each piece of lumber (total of 200 pieces) was determined. The Forest Products Laboratory treating plant has a 10-ft (3-m)-long cylinder that is capable of treating no more than 55 pieces of £10-ft (£3-m)-long 2 by 4 lumber. Because it was desirable to treat all the pieces in one charge to minimize variability. we decided to use 5-ft (1.5-m)-long test specimens and stack them end-to-end during treatment. Accordingly. each 12-ft (3.7-m)-long piece was cut 5 ft (1.5 m) long and the other material was discarded. In cutting the 5-ft (1.5-in) specimen. the grade-dictating defect was either centered over the entire length of the piece or located as close to midspan as possible.

Treatment

The borate-nitrogen, phosphate-free FR treating concentrate[3] was supplied by Osmose. Inc. [4] (Buffalo. NY). Prior FR testing had established a minimum chemical retention level for southern pine lumber to meet the required flame spread and flame progression limits set forth in national building codes and in AWPA Standard C 20 [5]. The experimental treatment employed a full-cell treatment process including a final vacuum. Two groups of 50 specimens were treated at one time. An initial vacuum of just over 25 in. Hg (–85 kPa) was held for 45 min; a 7% solution concentration of FR in water was then introduced into a 3-ft (0.9-m)-diameter. 10-ft (3-m)-long treating cylinder. Immediately afterward. 150 lb/in.^{2} (1 MPa) of pressure was held for 90 min. The treating solution was drained off at the end of the pressure period and a final vacuum of just over 25 in. Hg (–85 kPa) was held for 10 min. The average retention for the 100 treated lumber specimens was 2.57 lb/ft^3 (41.7 kg/m^3), and the standard deviation was 0.37 lb/ft^3 (6.0 kg/m^3).

Drying

The FR-treated specimens were kiln dried after treatment using a 2000-ft^3 (56-m^3) steam-heated brick kiln We used a four-step post-treatment kiln-drying schedule applicable to relatively small volumes of lumber in experimental-sized kilns and within the temperature limitations of AWPA Standard C20 (Table 1) The first two stages of this kiln-drying regime were intended to facilitate heat absorption by the wood the latter two stages facilitated drying The maximum kiln temperature of 160°F (71°C) was achieved at 48 h into the kiln drying process The total time in the kiln was 120 h. Initial lumber moisture content was 110 to 125%. and final moisture content was 15 to 18%. Throughout kiln drying. an average air speed of 200 to 220 ft/s (65 to 70 m/s) was maintained through the load, with fan reversal every 3 h While this schedule was appropriate for small kiln loads. different schedules using similar maximum temperature limits will be needed for commercial kilns.

[3] Fire-retardant formulation used as described in U.S. Patent No. 6,306,317.

[4] The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Department of Agriculture of any product or service. Osmose, Inc. is not associated with the Federal government The information given in this section should not be construed as an endorsement or approval of the chemical or processes reported.

TABLE 1— *Specific gravity and moisture content at time of test of FR-treated and end-matched untreated 2 by 4 southern pine lumber. a*

Treatment	Time at 150°F (66°C). Days	Specimens No.	Specific Gravity	Moisture Content. %
Untreated	0	49	0.49 (0.04)	12.1 (0.6)
	108	48	0.49 (0.05)	12.5 (0.5)
FR	0	50	0.51 (0.05)	13.9 (0 8)
	108	49	0 50 (0.04)	13.5 (0.5)

[a] Values in parentheses are standard deviation.

Exposure

After treating and redrying, all specimens (both untreated and FR-treated) were conditioned to constant weight at 74°F (23°C) and 65% RH After conditioning at these approximate 12% moisture content conditions. each 5-ft (1.5-m)-long 2 by 4 specimen intended for high-temperature exposure was exposed at 150°F (66°C) and 75% RH for 108 days in a Forma-Scientific™ environmental chamber After the exposure period. all specimens (untreated and FR-treated exposed and unexposed) were reconditioned to constant weight at 74°F (23°C) and 65% RH.

Testing

After conditioning. each 2 by 4 specimen was tested to destruction in a four-point bending test using three-point loading (ASTM D 5664 [4]) with a test span of 57.5 in (146 cm). The rate of loading was 1 in (2.54 cm) per minute. which caused failure in 1 to 3 min. Centerpoint deflection was measured using a linear variable differential transducer (LVDT) mounted on a yoke suspended from pins located at the neutral axis above the reaction supports. Load was measured with a calibrated load cell. Both the load cell and the LVDT were interfaced to a computer that recorded load and deflection Maximum load capacity (P_{max}) was obtained from the load data. Modulus of elasticity (*MOE*). modulus of rupture (MOR), and work to maximum load (*WML*) were calculated from measured sizes and from recorded load and/or deflection data After each specimen was tested to failure. a 3-in (76-mm)-long section of full width and thickness was cut from near the point of failure and used to calculate specific gravity and moisture content at time of test

Analysis

Data analysis took into account the *MOE* of the original 12-ft (3.7-m)-long 2 by 4 lumber that was obtained by transverse vibration. The tests of significance were obtained using analysis of covariance techniques employing the original transverse-vibrational MOE as the covariable. For each mechanical property, we made two preliminary comparisons. In the first comparison, we tested and evaluated the significance of the initial effect of treatment and redrying on lumber properties. In the second. we tested and evaluated the significance of any difference in the rate of property loss over time of high-temperature exposure. Although a 95% level of significance is sometimes considered the paramount criterion, employing a test at the 90% level of significance can indicate impor-

TABLE 2—*Properties of FR-treated and end-matched untreated 2 by 4 southern pine lumber.*

Property and Treatment	Time at 150°F (66°C). Days	n^a	Mean	SD	Value at Various Percentiles				
					10th	25th	50th	75th	90th
MOE (X 10^6 lb/in.^{2})									
Untreated	0	49	1.5	0.4	0.96	1.28	1.53	1.75	2.08
	108	48	1.7	0.5	1.05	1.26	1.72	2.1I	2.33
FR-treated	0	50	1.6	0.4	1.08	1.32	1.59	1.93	2.22
	108	49	1.9	0.5	1.12	1.50	1.80	2.17	2.5I
MOR (X 103 lb/in.2)									
Untreated	0	49	7.5	2.3	3.9	5.6	7.7	9.0	10.2
	108	48	7.9	3.1	3.8	5.6	7.7	10.3	12.6
FR-treated	0	50	6.8	2.7	3.8	4.9	6.2	8.8	11.3
	108	49	6.8	2.5	3.5	5.2	6.7	8.5	10.2
P_{max} (x 10^3 lbf)									
Untreated	0	49	2.4	0.8	1.2	1.8	2.5	2.3	3.3
	108	48	2.4	0.9	1.2	1.7	2.3	3.0	3.8
FR-treated	0	50	2.2	0.9	1.2	1.5	2.0	2.8	3.5
	108	49	2.1	0.8	1.0	1.6	2.0	2.6	3.0
WML (in·lb/in^3)									
Untreated	0	48	4.84	2.55	2.35	3.10	4.33	5.83	9.04
	108	48	4.72	2.85	1.71	2.75	3.77	6 65	8.97
FR-treated	0	49	3.45	2.89	1.25	1.78	2.50	4.40	6.01
	108	49	2.99	1.72	0.95	1.66	2.73	3.94	5.78

[a] Sample size

tant trends that may influence long-term performance. Thus, considering the limited sample size (50) required in Method C of ASTM D 5664, we employed a 90% level of significance as our criterion for significance.

Results

In some cases, mechanical property data were excluded from analysis, resulting in sample sizes of 50, 49, 49, and 48 for the four groups. In two cases, the data were not considered because the failure occurred outside the zone of uniform moment and true ultimate strength could not be calculated. In the other instances, the LVDT, which measures deflection during the bending tests, was either damaged or malfunctioned. This loss of data should not influence the practical interpretation of the experimental results.

Specific gravity and moisture content of each group at the time of testing are given in Table 1. The mean, standard deviation, and nonparametric (i.e., rank order) estimate of the 10th, 25th, 50th, 75th, and 90th percentiles for *MOE, MOR,* P_{max}, and *WML* are shown in Table 2. An estimate of the effects of treatment and high-temperature exposure was developed by calculating the ratio of each property estimate for each treated or exposed group and its matched estimate for the untreated, unexposed control These ratio-based comparisons are shown in Figs 1 to 4. The *p*-values and tests of least-square means (means adjusted to common initial MOE value) at the 90% level of significance are shown in Table 3. Because the initial sorting procedure systematically assigned comparable proportions of high, medium, and low MOE specimens to each group, the adjusted means were quite similar to the unadjusted means. The results of these tests indicate important differences between treated and untreated material.

Note that while *MOR* and P_{max} are related, they are not related by a simple constant. Treatment normally causes a finite amount of irreversible swelling in direct relation to the properties of the treatment chemical and amount absorbed. This swelling causes increases in specimen width (*w*) and thickness (*b*) and affects the de-

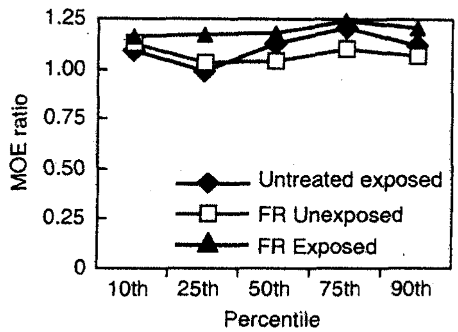

FIG. 1—*Ratio of modulus of elasticity (MOE) of treated and exposed 2 by 4 southern pine lumber and MOE of matched untreated, unexposed controls across distribution for each treatment-temperature exposure group.*

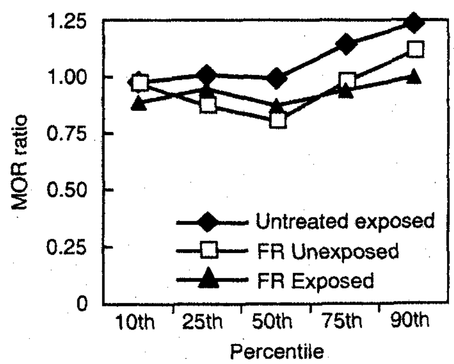

FIG. 2—*Ratio of modulus of rupture (MOR) of treated and exposed 2 by 4 southern pine lumber and MOR of matched untreated, unexposed controls across distribution for each treatment–high-temperature exposure group.*

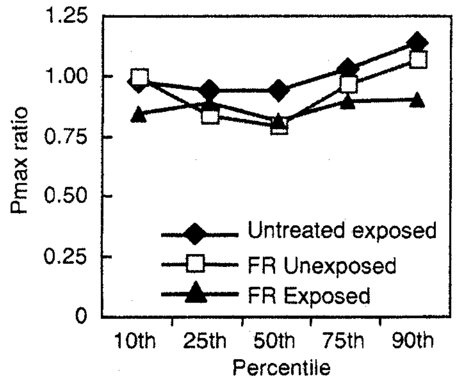

FIG. 3—*Ratio of maximum load capacity (P_{max}) of treated and exposed 2 by 4 southern pine lumber and P_{max} of matched untreated, unexposed controls across distribution for each treatment-high-temperature exposure group.*

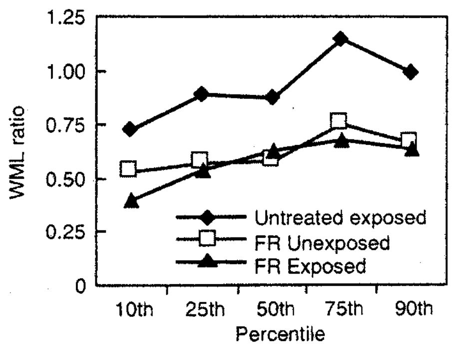

FIG 4—*Ratio of work to maximum load (WML) of treated and exposed 2 by 4 southern pine lumber and WML of marched untreated, unexposed controls across distribution for each treatment-high-temperature exposure group*

TABLE 3—*Significance of equality in means of all groups, initial property effect* (b_0), *and rate of property change* (b_1)[a].

Property	H_0: Equality Test of Four Means	H_0: β_U-β_{0T}	H_0: β_{1U}-β_{1T}
MOE	0.0001	0.276	0.399
	U0 TO UE TE	NS	NS
MOR	0.1000	0.187	0.594
	TO TE U0 UE	NS	NS
P_{max}	0.1360	0.121	0.938
	TE TO UE U0	NS	NS
WML	0.0003	0.006	0.638
	TE TO UE U0	Significant	NS

[a] Probability (p) values derived by analysis of covariance for individual mean parameter differences.

H_0, equality test of mean values derived for untreated-unexposed (U0), treated-unexposed (T0), untreated-exposed (UE), and treated-exposed (TE). Lines represent equality of mean property at 90% level of significance. NS means not significant.

H_0: β_{0U}-β_{0T}, test of equality of property for untreated-unexposed and treated-unexposed groups.

H_0: β_{1U}-β_{1T}, test of equality of rate of change in property for untreated and treated material.

pendence of *MOR* to P_{max} according to its nonlinear effect on the relationship:

$$MOR = 2P_{max} \cdot span/(b \cdot w^2) \quad (1)$$

While P_{max} is simply a load value. note that MOR is affected by changes in material size (i.e., swelling). Thus, most waterborne treatments, such as the FR tested in this study. will induce moderate swelling. This could result in noticeable differences between the observed effects between MOR and P_{max}. For treatments that do not cause measurable swelling. MOR and P_{max} would be similar. For treatments that do cause swelling, P_{max} might be a more reliable indicator of the effect of treatment on design.

Discussion

As expected, the results showed a slight increase in specific gravity of FR-treated southern pine lumber, which directly reflects the weight of absorbed FR chemical in the lumber. We also noted a lack of change in specific gravity after extended high-temperature exposure, which indicates an apparent lack of differences in the loss in wood density for either FR-treated or untreated lumber after 108 days of exposure to 150°F (66°C).

The actual initial (i.e., no exposure) difference in MOE between FR-treated and untreated southern pine lumber was about 5% (Table 2), but this initial difference was not significant (Table 3) and was generally uniform across the entire MOE distribution (Fig. 1), Both FR-treated and untreated lumber experienced a slight increase in MOE after the exposure period. However, a test for difference in the rate of change in MOE showed no significant differences in the rate of change between FR-treated and untreated material (Table 3).

The results from Method C of ASTM D 5664 indicated an initial difference of approximately 8% between the bending strength (i.e., MOR) values of matched groups of FR-treated and untreated 2 by 4 southern pine lumber (Table 2). However, this initial difference was not found to be significant at a £ 0.10 (Table 3). When compared to untreated controls exposed at 150°F (66°C) for 108 days, 2 by 4s treated with FR and exposed to high temperature showed an approxiinate 13% decrease in *MOR* (Table 2). This difference between untreated and FR-treated specimens, both exposed at 150°F (66°C) for 108 days, was significant at a £ 0.10 (Table 3). The measured initial treatment effect of an 8% decrease in *MOR* is less than that found in previous tests of other phosphate-based fire retardants; the post-exposure effect of a 13% decrease in *MOR* is approximately half that found in previous tests [6–10].

Unlike for *MOE,* the effects of various combinations of treatment and high-temperature exposure were not consistently uniform across the *MOR* distribution (Fig. 2). Nevertheless, no practical difference in the effect of treatment-exposure on *MOR* as a function of wood quality should be inferred. This opinion is based on experience from analyzing the results of many previous treatment-effects studies and especially experience with the variability of property estimates for small groups of lumber of approximately this sample size (50 specimens). We believe the trend of an increase in *MOR* ratio might be related to irreversible lowering of the moisture holding capacity of the wood (Fig. 2). The variability in MOR of untreated and FR-treated lumber exposed for 108 days at 150°F (66°C) was noticeably less than the variability in MOR of matched unexposed lumber (Table 1). Several researchers have noted such an irreversible loss in hygroscopicity after wood is exposed to elevated temperatures for extended periods [6,8–11]. This loss in hygroscopicity might also tend to affect stronger specimens more than weaker material because the strength of specimens might be dictated by clearwood strength rather than knots or defects. In analyzing the effects of long-term exposure at 150°F (66°C), we found that FR-treated lumber showed no significant differential rate of loss in *MOR* when directly compared to the *MOR* of matched untreated lumber (a £ 0.187) (Table 3),

The actual difference in load capacity (P_{max}) between FR-treated and untreated southern pine lumber not exposed to high temperature was about 9% (Table 2). This difference was not significant at a £ 0.10, but from a practical aspect its validity did border on significance (a £ 0.121) (Table 3). Based on previous experience. our opinion is that the effect of treatment and long-term high-temperature exposure on P_{max} should generally be considered uniform across the P_{max} distribution (Fig. 3). The rate of change in load capacity (i.e., the loss of load-carrying capacity from long-term exposure to elevated temperatures) was not significantly different between FR-treated and untreated material. Based on our experience with earlier generations of boron-nitrogen FR formulations [6, 9, 11], these results of strength and load-capacity effects were not unexpected.

The earlier borax/boric acid-based formulations have not caused significant strength loss. nor have they caused significant changes in strength loss over time of exposure at high temperature when compared to the strength of untreated wood [6, 7, 10]. However. these formulations have caused brashness (e.g., embrittlement) in FR-treated material. which seemingly affects fracture mechanisms and the ductility of wood treated to high borate retentions. A basic assumption of wood engineering design is that wood will often react like a ductile material rather than a brittle material. The fact that previous boron-nitrogen FR formulations embrittled the treated wood appears to violate this assumption.

Our results were empirically similar to previously reported results for earlier borax-boric acid-based formulations. Work to maximum load of treated lumber was significantly reduced (by about 29%) coinpared to that of matched untreated lumber (Table 3). The loss in energy absorption was slightly greater for low strength material than for high strength material (Fig. 4). However, the rate of loss in *WML* on long-term high-temperature exposure was not significantly different between FR-treated and untreated material. Possibly most important, no visual changes in the appearance o characteristics of the fracture surfaces were noticeable in direct comparisons between FR-treated and untreated lumber. Thus. while the reduction in *WML* should be directly accounted for in engineering design. these results of unchanged fracture mechanisms provide support for the application of simple and direct engineering adjustment factors rather than major alterations in engineering practice because of concerns of increased brittleness.

One objective of our research was to critique the D 5664-95 test method. The reported results for full-size 2 by 4 lumber clearly parallel the results obtained previously with small clear specimens cut from 2 by 4 lumber. Little additional inforination was gained by performing both series of tests (i.e., both Method B and Method C evaluations). As concluded in Part II of this series. cutting small clear specimens from a species like white spruce. with its many small knots. was extremely difficult. We recommend modifying ASTM D 5664 so that it requires evaluation of both the initial effect of FR treatment and the potential in-service effect of exposure to elevated temperatures on just two species of full-size lumber (southern pine and Douglas-fir) rather than evaluation of three species of small clear specimens cut from lumber. Cutting the number of small clear specimens currently required by Methods A and B of ASTM D 5664 is very expensive with respect to labor and virtually impossible with wine wood species Most important the application of results derived in Methods A and B from tests of mall clear specimens to full-size dimension lumber requires assumptions that would not be necessary if we simply tested the full-size lumber

Conclusions

The data and trends reported here are indicative of results expected from fire-retardant (FR)treatment retentions and post-treatment kiln temperatures similar to those evaluated in this project. When tested according to Method C of the ASTM D 5664-95 standard test method, the results indicate that treatment of nominal 2 by 4. No. 1 grade. southern pine lumber with the new boron-nitrogen, phosphate-free FR may reduce some mechanical properties. Treated and redried lumber experienced an insignificant (a £ 0.187) 8% loss of bending strength when compared to strength before exposure at 150°F (66°C). After 108 days of exposure at this temperature. treated and redried lumber experienced a significant (a £ 0.10) 13% loss of bending strength. Accordingly, potential engineering design adjustments of −8% for the initial effect on bending strength or - 13% for the combined initial and post-exposure effect may be reasonable. Treated and redried lumber experienced a marginally significant (a £ 0.121) loss in maximum load capacity of about 9%. Treated lumber also experienced a significant loss (29%)in work to maximum load. No significant negative initial treatment effect occurred in modulus of elasticity.

An analysis for the potential of secondary treatment effects related to thermal degradation from in-service exposure to elevated temperatures showed no indication that the FR-treated lumber would experience accelerated thermal degradation when compared to matched untreated lumber exposed to elevated temperature.

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