FLAME RETARDANCY

Session 5
TESTING/STANDARDS/METHODS

5-A CONE CALORIMETER EVALUATION OF WOOD PRODUCTS

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ABSTRACT

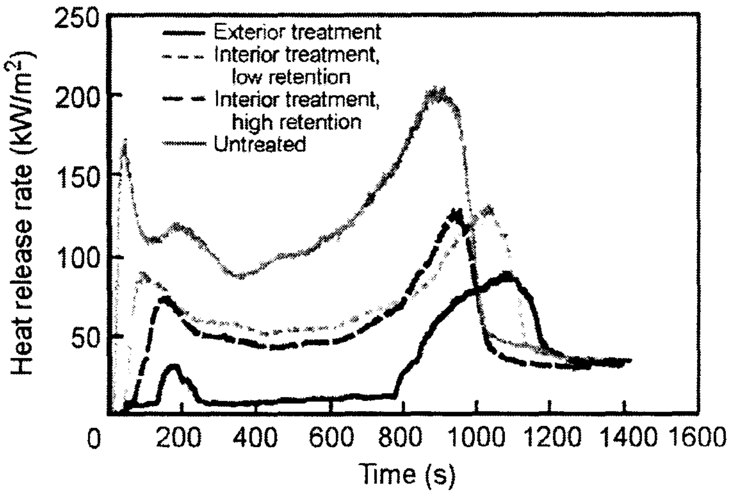
The Forest Products Laboratory uses the cone calorimeter for the initial evaluation of the flammability of untreated and fire retardant treated wood products. The results of various studies are reviewed using a model presented at the 12th Annual BBC Conference on Flame Retardancy. The model uses data from the cone calorimeter to provide measures of fire growth propensity based on material bulk properties and surface fire growth propensity and to provide an estimate of the ASTM E 84 flame spread index. In a study on the fire performance of treated wood exposed to elevated temperature, wood treated with borax/boric acid did not maintain fire retardancy after being exposed to 66°C (150°F) for 1 to 3 months.

INTRODUCTION

At the Forest Products Laboratory, USDA Forest Service, the cone calorimeter has replaced other test methods for initial evaluation of the flammability of untreated and treated wood products. In our early use of the cone calorimeter, test results such as the peak heat release rate (PHRR) and the 300-s average heat release rate (HRR) were used to estimate the ASTM E 84 flame spread index. In a 2001 paper, a predictive methodology was presented that uses cone calorimeter results to provide an overall evaluation of fire growth propensity. For fire retardant treated (FRT) wood products, we also use the model to estimate the ASTM E 84 flame spread index. This methodology incorporates the time to ignition, PHRR, and total heat release rate from the cone calorimeter test. In this paper, we review some results from applying the model to investigating the effect of elevated temperature on the fire performance of wood treated with a borax/boric acid treatment or monoammonium phosphate.

FIGURE 1

HEAT RELEASE RATE (HRR) CURVES FOR UNTREATED AND TREATED SOUTHERN PINE



CONE CALORIMETER

In the cone calorimeter test, a 100-mm square specimen is exposed to a constant external heating flux. The primary result is an HRR curve over the duration of the test. The HRR due to combustion is determined using oxygen consumption methodology, which is derived from the observation that the net heat of combustion is directly related to the amount of oxygen required for combustion. Thus, the test measures oxygen concentration and exhaust gas flow. A typical curve for wood is an initial increase to a PHRR, a drop to a steady-state HRR, and a second peak as the final portion of the specimen is consumed (Fig.1). A very small or non-existent first peak is sometimes observed with very good fire retardant treatments [1]. For reporting purposes, the HRR curve is often reduced to single numbers via the initial PHRR and averages of the HRR over a set time (60, 180, and 300 s) after ignition of the specimen. HRR is measured in kilowatts per square meter (kW/m^2). The total heat release (THR) is the cumulative heat release (area under the heat release curve) over the duration of the test. The cone calorimeter method is described in ASTM E 1354–02 [2] and ISO 5660–1 [3].

The mass loss of the burning specimen is also recorded in the cone calorimeter test. From the heat release and mass loss, the effective heat of combustion (EHOC) (heat release per unit mass loss) is calculated. The average effective heat of combustion (AEHOC) is computed from THR divided by total mass loss. The obscuration of a laser beam in the exhaust duct is recorded as a measure of the visible smoke development from the burning specimen. The average specific extinction area (ASEA) is computed from the smoke obscuration data. Ignitability is determined by observing the time for sustained ignition of the specimen.

In general, we test in the horizontal orientation, with the conical radiant electric heater located above the specimen and the retainer frame (without the wire grid) over the test specimen. The electric spark igniter is placed above the test specimen until sustained ignition is observed. Heat flux levels of 35 or 50 kW/m^2 are used to test wood products. For the predictive model discussed in this paper, an external heat flux of 50 kW/m^2 was used to obtain the cone calorimeter test data. HRR is a critical factor in the spread of flames over a surface and the overall growth of a compartment fire. It is an option for evaluating the degree of combustibility of different materials. Fire-retardant treatments for wood products are designed to reduce their flammability. In the United States, the regulatory test for flammability of building products is the 7.32-m (25-ft) tunnel test [4].

PREDICTIVE MODEL

The cone calorimeter has been used to provide estimates of the flame spread index (FSI) obtained in the tunnel test [4]. In earlier work with an Ohio State University HRR apparatus (ASTM E 906), we used the 300-s average HRR to estimate the FSI [5]. Dietenberger and White [6] developed a fire growth propensity model useful for materials with an FSI of 70 or less.

The model development assumes an exponentially decaying material HRR profile as a function of time after initial ignition and of initial ignited area. The materials are assumed to behave as thermally thick materials at a flux of 50 kW/m^2. The THR assumes that the specimen was completely charred for the material thickness δ. A $(12.5/\delta)$ parameter in the equations adjusts the numbers to the assumed completely charred 12.5-mm-thick sample used in the model development.

The model uses PHRR, THR, and time for sustained ignition from the cone calorimeter test to provide estimates for the ASTM-E-84 FSI. An acceleration parameter, ß, is calculated from the PHRR ($\dot{Q}_P$, kW/m^2) from the cone calorimeter test, an exponential time decay coefficient (ω), and a material time constant (τ). The acceleration parameter, derived from analytical flame spread formulas, is

$$\beta = b + c\dot{Q}_P - \varpi\tau \qquad (1)$$

For the ASTM E 84 tunnel test, the coefficients *b* and *c* have values of -0.085 and 0.00188, respectively. These coefficients were obtained by fitting the flame spread model to empirical data. The exponential time decay coefficient, w, is given by:

$$\varpi = \dot{Q}_P / \{1000 Q_T (12.5/\delta)\} \qquad (2)$$

where Q_T is THR (MJ/m^2) from the cone calorimeter test and δ is specimen thickness (mm). The material time constant τ is given by

$$\tau = (4/\pi) t_i \qquad (3)$$

where t_i is time for sustained ignition (s). The term

$$\gamma = \{Q_T (12.5/\delta)\} / (4t_i/\pi) \qquad (4)$$

is a measure of fire growth propensity based on material bulk properties, while PHRR provides a measure of the surface fire growth propensity. Plotting γ on the vertical axis and PHRR on the horizontal axis provides a picture of the behavior of the material (Fig. 2). Low fire growth propensity can be due to either very low γ (y-axis) (low THR and high times for sustained ignition) or very low PHRR. An acceleration parameter above zero indicates a degree of fire growth above that of steady growth. A degree of damping of fire growth after ignition is indicated by negative values. The curves for ß of zero, 0.184, and -0.1 are shown in Figure 2. Based an experimental regression, the following curve for the accelerating parameter and FSI was developed (Fig. 3):

$$\beta = 0.6[1 - \exp(-0.032\mathrm{FSI})] - 0.36 \qquad (5)$$

FIGURE 2

INITIAL FIRE GROWTH PROPENSITY OF PINE TREATED WITH COMMERCIAL FIRE RETARDANT TREATMENTS

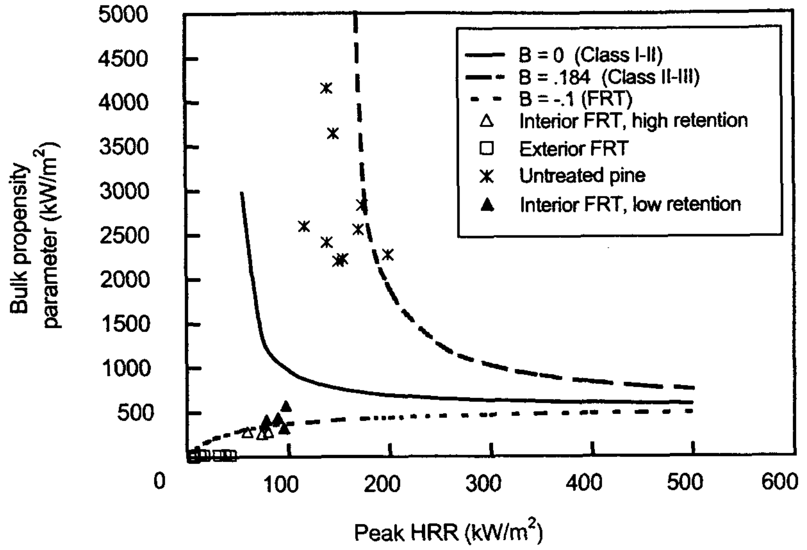
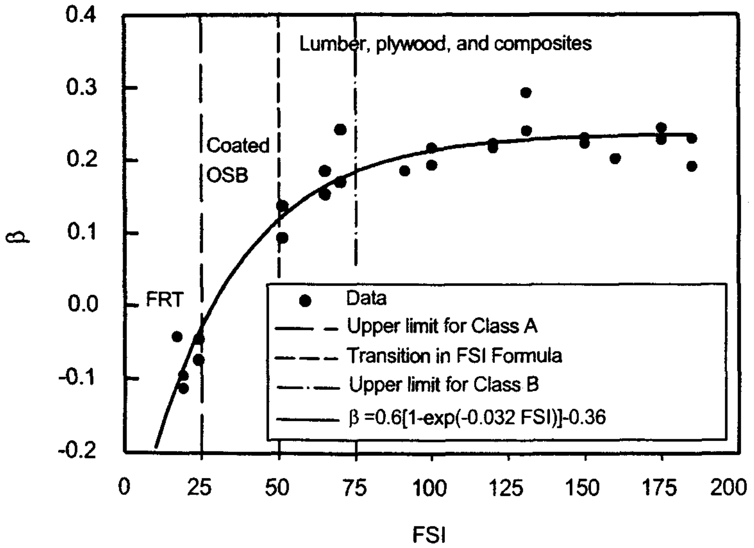


FIGURE 3

REGRESSION OF DATA USED TO DEVELOP PREDICTIVE EQUATION FOR ASTM E 84 FSI (EQ. 6)



The predictive equation for FSI becomes

$$FSI = -31.25\ln(0.4 - 1.67\beta) \qquad (6)$$

FRT wood with Class I FSI (≤ 25) will have data points around the ß = –0.1 curve (FSI~18). Products with Class II FSI (26 < FSI ≤ 75) will have data points between the ß = 0 curve (FSI~29) and the ß = 0.184 curve (FSI~74). Untreated wood with Class III FSI will have data points greater than the ß = 0.184 curve. As Figure 3 indicates, the equation for estimating FSI is not sensitive to variations in FSI > 75 or < 25. Data for the standard test duration of 10 min, as specified in E 84, were used to obtain the coefficients in the equation. For classification as FRT wood, the normal duration of the tunnel test is increased to 30 min.

TREATED SOUTHERN PINE

Southern Pine samples were pressure treated with a commercial interior or exterior treatment (Fig. 1).* External heat flux was 50 kW/m^2, and the retainer frame (without the wire grid) was placed over the test specimen. The data for the interior treatment were for two chemical retention levels. Test results used to obtain the fire growth propensity graph (Fig. 2) and the estimates for the ASTM E-84-FSI were PHRR, THR, and time for sustained ignition (Table I).

For the exterior treatment, the observed times for sustained ignition were considerably longer than the times for the untreated or interior treatments. These longer times corresponded with the second peak in the HRR curves (Fig. 1). The initial PHRR was not sufficient for observable sustained ignition. Visual observation of ignition can be difficult with fire-retardant treatments. An alternative criterion for ignition of 30-kW/m^2 HRR has been suggested [1]. The alternative comparative cone calorimeter results are 300-s average HRR, AEHOC, and residual mass percentage (Table I). These indicators are consistent with the trends of the FSI results.

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* Samples were tested for Professor Michael Barnes of the Forest and Wildlife Research Center, Forest Products Laboratory, Mississippi State University. The overall study was on FRT wood for repair of covered bridges and was funded by the U.S. Department of Transportation.

TABLE 1

CONE CALORIMETER RESULTS FOR SOUTHERN PINE

Sample	Est. FSI[a]	PHRR[b] (kW/m²)	THR (MJ/m²)	Time for ignition[c] (s)	300-s HRR[d] (kW/m²)	AEHOC[e] (MJ/kg)	RMF[f] (%)	ASEA[g] (m²/kg)
Untreated	60	154	142	28	117	11.9	0.19	78
FRT interior[h]								
Low	15	88	83	107	62	7.8	0.31	18
High	10	72	65	118	49	7.0	0.33	11
FRT exterior	−15	19	32	938	29	3.1	0.34	53

[a] Estimated ASTM-E-84 FSI calculated from peak heat release rate (PHRR), total heat release (THR), and time for sustained ignition.
[b] Initial PHRR.
[c] Visual observation of sustained ignition.
[d] Average HRR for 300 s after observation of sustained ignition.
[e] THR divided by total mass loss.
[f] Residual mass fraction.
[g] Average for duration from observed ignition to end of test.
[h] Low retention = 38 kg/m³; high-retention = 70.5 kg/m³.

The increase in residual mass fraction (Table I) has long been used to evaluate FRT wood. It is the basis for the fire tube test (ASTM E 69 [7]) and thermal gravimetric analysis (TGA) testing. Mass loss is also the basis for the new screening test that uses the conical heater and load cell of the cone calorimeter (ASTM E 2102 [8]).

To further illustrate the application of the model, we applied the model to data obtained in a study of the effect of elevated temperatures on the fire performance properties of FRT wood.

EFFECT OF ELEVATED TEMPERATURES

The effect of elevated temperatures on mechanical properties of FRT wood was extensively studied as a result of structural failures in the field [9,10], and ASTM D 5664 [11] and related standards were consequently developed. At that time, studies were not being conducted on the effect of the elevated temperature on the fire performance of treated wood. In a subsequent small, unpublished study, samples were treated with simple inorganic salts, exposed to 66°C (150°F), and tested in the cone calorimeter.; Two of the treatments evaluated were monoammonium phosphate and a mixture of borax/boric acid.

* Study conducted by Mitchell Sweet and Melissa Hill at the Forest Products Laboratory; statistical analysis provided by Cherilyn Hatfield.

The pine and maple wood samples were 16 mm thick. Samples were treated to a dry chemical retention of 58 kg/m^3. Selected samples were then heated at 66°C for 1, 3, and 6 months. Cone calorimeter tests were also conducted on untreated and treated samples kept at room temperature for 0, 1, 3, and 6 months. Samples were tested without the retaining frame or grid, and the heat flux was 50 kW/m^2. Three replicates were tested.

Without exposure to elevated temperature, samples treated with these two well-known fire retardants exhibited HRR curves that were significantly lower than that of the untreated samples. Representative HRR curves for pine samples treated with the borax/boric acid mixture are shown in Figure 4. Samples treated with the borax/boric acid mixture had higher HRR values after 1 month of exposure to 66°C. Figure 5 illustrates the increased fire growth propensity of the borax/boric acid samples exposed to 66°C for 1, 3 and 6 months. For the samples treated with monoammonium phosphate, we detected no significant increase in HRR after 6 months of exposure at a constant temperature of 66°C. The fire performance of these samples was unaffected by the elevated temperature (Fig. 6). After 6 months, the estimated FSI of the maple samples treated with borax/boric acid had increased from 29 to 66 and that of the pine samples from 25 to 67 (Fig. 7 and Table 2).

For the borax/boric acid samples, the elevated temperature exposure affected each variable used to calculate the estimated FSI (Table 2). Elevated temperature also adversely affected 300-s average HRR, residual mass fraction, AEHOC, and ASEA (Table 2).

FIGURE 4

HRR CURVES FOR PINE TREATED WITH BORAX/BORIC ACID MIXTURE AND EXPOSED TO 66°C FOR 0, 1, AND 6 MONTHS

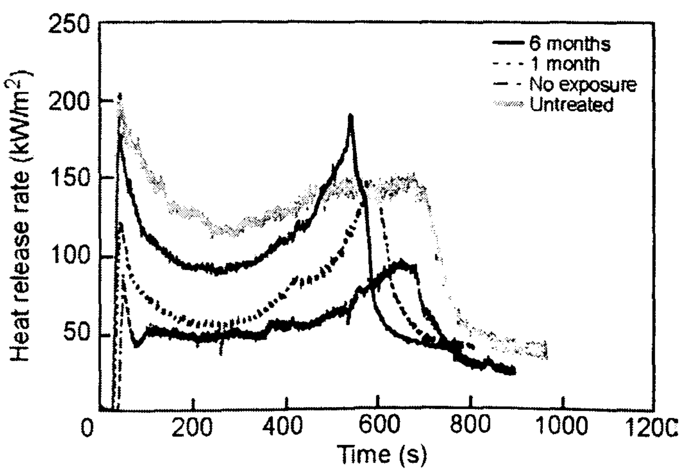

FIGURE 5

FIRE GROWTH PROPENSITY OF PINE TREATED WITH BORAX/BORIC ACID MIXTURE

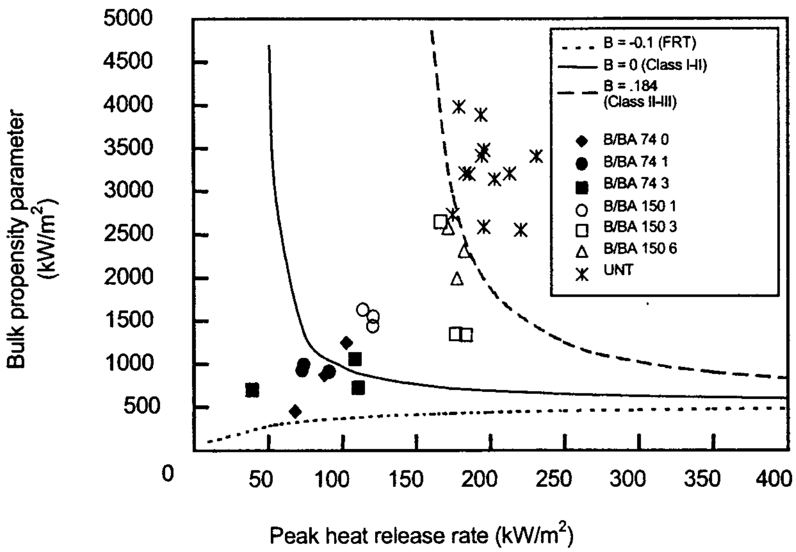

FIGURE 6

FIRE GROWTH PROPENSITY OF PINE TREATED WITH MONOAMMONIUM PHOSPHATE

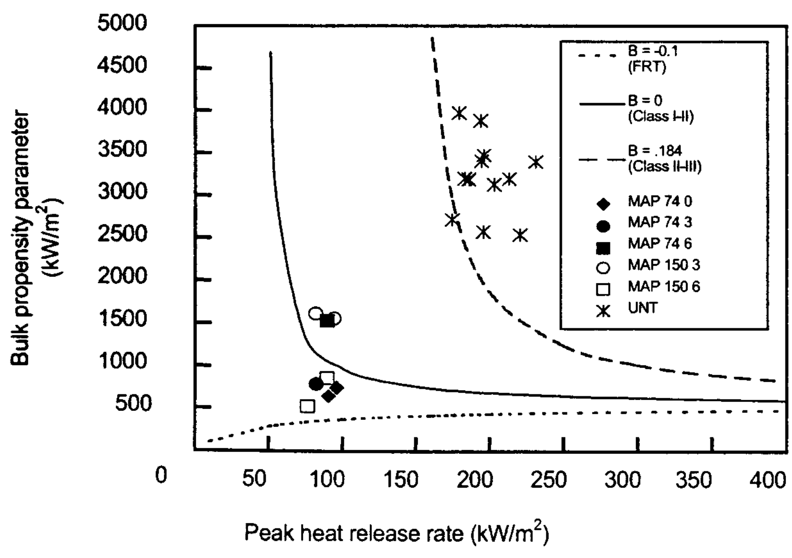

FIGURE 7

EFFECT OF ELEVATED TEMPERATURE ON ESTIMATED FSI FOR PINE TREATED WITH BORAX/BORIC ACID MIXTURE AND MONOAMMONIUM PHOSPHATE

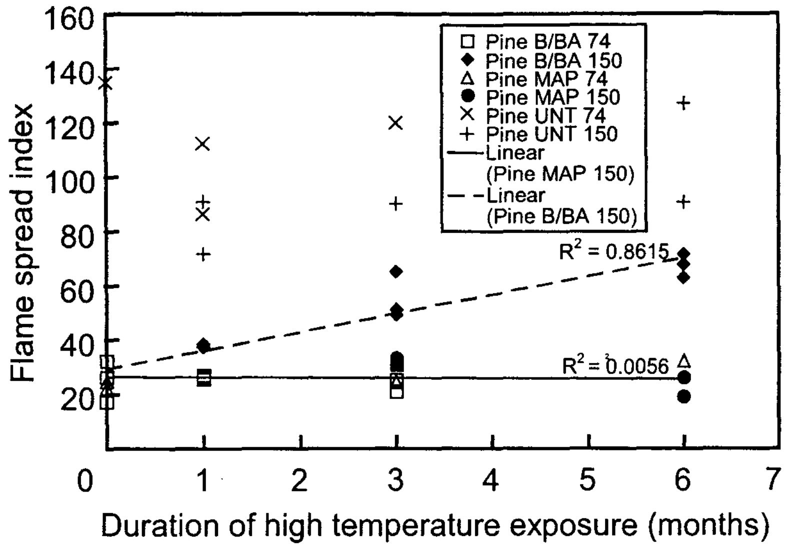

TABLE 2

CONE CALORIMETER RESULTS FOR BORAX/BORIC ACID TREATED SOUTHERN PINE EXPOSED TO ELEVATED TEMPERATURE (66°C)[A]

Sample	Est. FSI	PHRR (kW/m²)	THR (MJ/m²)	Time for ignition (s)	300-s HRR[d] (kW/m²)	AEHOC (MJ/kg)	RMF (%)	ASEA (m²/kg)
Pine								
Untreated	>72	208	106	30	138	12.44	0.18	65.44
Treated								
0 months	25	86	44	52	50	6.54	0.37	4.27
1 month	38	119	53	31	68	8.03	0.34	5.24
3 months	55	176	63	35	88	9.33	0.30	17.13
6 months	67	177	71	28	108	9.42	0.28	29.91
Maple								
Untreated	>80	209	114	29	146	12.02	0.18	50.14
Treated								
0 months	29	85	56	43	64	7.15	0.34	2.77
1 month	45	148	68	39	82	7.87	0.27	8.92
3 months	62	183	83	42	101	9.50	0.26	21.14
6 months	66	177	80	33	103	9.23	0.25	37.17

[a] See footnotes to Table 1 for definition of terms.

Statistical analysis of ignition time, PHRR, and average HRR data confirmed that differences between samples heated at 66°C and those left at room temperature were statistically significant for the borax/boric acid treatment but not for monoammonium phosphate. This fire performance behavior is in sharp contrast to Forest Products Laboratory research on mechanical properties of FRT wood in which the losses in mechanical properties of borax/boric acid treated wood were significantly less than losses observed for wood treated with monoammonium phosphate when exposed to an elevated temperature of 66°C.

In fact, treatment with boron is advocated as a solution for treatments that adversely affect the mechanical properties of wood when exposed to elevated temperatures [12,13]. Further study is needed to identify the specific mechanisms of the reduction in fire retardancy and the forms of boron treatments that exhibit similar behavior. In the kiln drying of boron-treated wood, a major problem is the migration of salts to the wood surface and their loss into the atmosphere [14].

CONCLUSION

A previously developed model for predicting the ASTM E 84 flame spread index from cone calorimeter data was reviewed. The regression of the model parameter and the flame spread index can benefit from further refinements that will come with additional comparative data. The model is useful in comparing the relative performance of treated wood products. Examples of test results for untreated and treated wood were provided.

In a study on the fire performance of treated wood exposed to elevated temperature, wood treated with borax/boric acid did not maintain its fire retardancy after being exposed to 66°C. Further research is needed on the mechanisms of the loss of fire retardancy. As with mechanical properties, the fire retardancy of FRT wood should be tested after exposure to periods of elevated temperature.

ACKNOWLEDGMENT

The cone calorimeter tests reported in this paper were conducted by Anne Fuller, technician in fire research unit at the Forest Products Laboratory.

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