Fire Performance Testing of WPCs at the Forest Products Laboratory

Nicole M. Stark*, Robert H. White

USDA Forest Service, Forest Products Laboratory, One Gifford Pinchot Drive, Madison, Wisconsin 53726, USA

Abstract

Wood-plastic composites represent a growing class of materials used by the residential construction industry and the furniture industry. For some applications in these industries, the fire performance of the material must be known, and in some cases improved. Lab-scale fire performance tests allow for comparisons between WPC formulations. At the Forest Products Laboratory (FPL) oxygen index tests and cone calorimetry are typically used during fire performance evaluations of WPCs. This paper describes these tests and presents a case study applying these methods for evaluation of wood flour-polyethylene composites containing fire retardants. In the case study we compared the results of wood-polyetheylene composites with unfilled polyethylene and solid wood and then evaluated the effect of five additive-type fire retardants on fire performance. Using these test methods we could identify the most promising fire retardants for wood-polyethylene composites.

Introduction

Wood-plastic composites (WPCs) represent an emerging class of materials that combines the favorable performance and cost attributes of both wood and plastics. Due to these attributes, forest-products companies see WPCs as a way to increase the value-added utilization of waste wood and wood of low commercial value. Plastic processors see wood as a readily available, relatively inexpensive filler that can lower resin costs, improve stiffness, increase profile extrusion rates, and act as an environmentally friendly way to decrease the use of petroleum-based plastics [1].

Currently several commercial WPCs are manufactured for the residential construction industry, primarily as lumber for decking and railing systems as WPCs offer a “wood” look and feel with minimum maintenance. Manufacturers are also introducing new applications for the furniture industry. Further expansion into the residential construction industry and development of applications for the furniture industry require an understanding of the fire performance of WPCs. Through lab-scale fire performance tests conducted at the FPL we are able to compare various formulations of WPCs and then evaluate the influence of formulation on fire performance.

Lab-Scale Fire Performance tests for WPCs at FPL

To evaluate the fire performance of WPCs at the FPL, we typically use two different lab-scale test methods. The first method is the oxygen index test. The objective of this test is to determine the minimum concentration of oxygen that will just support flaming combustion in a

mixture of oxygen and nitrogen. This test method is conducted according to ASTM D 2863, the Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index) [2]. During this test a sample is mounted vertically with support at the bottom. A transparent chimney is placed over the sample and oxygen and nitrogen flow upwards through the chimney. The top of the specimen is ignited and the burning behavior is observed. Figure 1 shows a WPC sample burning in an oxygen index apparatus. By changing the oxygen to nitrogen ratio, the minimum oxygen concentration for combustion is determined. This is reported as the oxygen index (OI). A higher OI is an indicator of possible better fire performance in terms of flame retardancy. As noted in the ASTM standard [2], correlation of the oxygen index with burning characteristics under actual use is not implied. The OI test is widely used in the evaluation of flame retardant treatments for plastics. We do not use the oxygen index test to test treatments for solid sawn wood.

The second test that we typically conduct for both WPC and other wood products is cone calorimetry. In this technique we measure the response of WPCs exposed to controlled levels of radiant heating. We conduct this test according to ASTM E 1354, the Standard Test Method for Heat and Visible Smoke Release Rates for Materials and Products Using an Oxygen Consumption Calorimeter [3]. In this test method, WPCs samples are burned in ambient condition while being subjected to a predetermined external heat flux. The primary result from the cone calorimeter test is a heat release rate (HRR) versus time curve. HRR is defined as the heat evolved from the specimen per unit time, and is determined by the oxygen consumed during burning [3]. Heat evolved from a material can contribute to the temperature of the environment and the rate of fire spread. Therefore, it is desirable for a material to have a lower HRR to decrease the contribution to a fire. Also recorded is ignitability, determined by observing the time for sustained ignition of the specimen and reported as ignition time (IT). For reporting purposes, the heat release curve is often reduced to single values via the recorded initial peak HRR and calculated averages of the HRR over a set time after ignition of the specimen was observed. The total heat release (THR), i.e., the cumulative heat release over the duration of the test, the average mass loss rate (MLR), and average effective heat of combustion (EHOC) for the test duration can also be determined according to ASTM D 1354 [3]. Figure 2 shows a WPC exposed to a conical heater during cone calorimetry.

Figure 1. WPC burning during an oxygen index test.

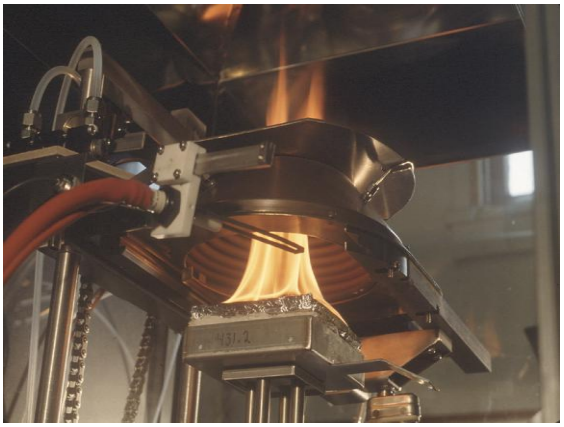

Figure 2. WPC burning during a cone calorimetry test.

Application: "Evaluation of Various Fire Retardants for Use in Wood Flour/Polyethylene Composites"

Oxygen index and cone calorimetry were used to evaluate various classes of fire retardants for use in wood-plastic composites. The main goal of this research was to create a baseline of information reporting fire performance of WPCs and the effectiveness of additive-type fire retardants [4].

Experimental methods

Materials and Manufacturing Method

The base WPCs investigated consisted primarily of polyethylene (PE) and wood flour (WF). The PE had a 5 melt flow index and was purchased from ExxonMobil (HD 6605.70, Houston, TX). American Wood Fibers supplied 40 mesh, mixed pine WF (AWF 4020, Schofield, WI). To maintain good composite surface characteristics, a lubricant was added to each composite. Struktol Company of America supplied the lubricant (TPW 113, Stow, OH). In addition, five fire retardant systems were investigated:

1) Decabromodiphenyl oxide (Saytex 102E, Albemarle Corporation, Baton Rouge, LA) and antimony trioxide (BrightSun HB, China Antimony Chemicals Co., Ltd., Guangxi, China)
2) Magnesium hydroxide (Magnifin H-10, Albemarle Corporation, Baton Rouge, LA)
3) Zinc borate (FireBrake ZB, Rio Tinto Minerals, Valencia, CA)
4) Melamine phosphate (Melapur MP, Ciba Specialty Chemicals, Tarrytown, NY)
5) Ammonium polyphosphate (Exolit AP 422, Clariant Corporation, Charlotte, NC)

The formulations examined are shown in Table 1. Composites without fire retardants had either 50% or 60% by weight WF. Composites with fire retardants incorporated 50% WF and 10% of the fire retardant system. This allowed composites to be compared based on WF content (50%) or PE content (35%). Unfilled PE samples were also manufactured.

Table 1. Formulations of WPCs manufactured with fire retardants.

	Composition based on weight (%)								
Code	**PE**	**WF**	**BR**	**AT**	**MH**	**ZB**	**MP**	**AP**	**Lub**
PE	100								
WF-50	45	50							5
WF-60	35	60							5
WF-BR	35	50	7.5	2.5					5
WF-MH	35	50			10				5
WF-ZB	35	50				10			5
WF-MP	35	50					10		5
WF-AP	35	50						10	5

PE = Polyethylene; WF = wood flour; BR = decabromodiphenyl oxide; AT = antimony trioxide; MH = magnesium hydroxide; ZB = zinc borate; MP = melamine phosphate; AP = ammonium polyphosphate; Lub = lubricant

A 32-mm Davis Standard (Pawcatuck, CT) twin-screw co-rotating extruder combined with a Schenck AccuRate (Whitewater, WI) loss-in-weight feeder system was used for all compounding. The barrel of the extruder had 10 separate zones, with zones 4 and 9 vented to the atmosphere. The screw had a 36:1 L/D ratio consisting of primarily of conveying elements, with kneading and mixing elements incorporated into the screw before the vents to build up pressure and disperse and mix the components. The extruder was outfitted with a strand die; the strand extrudate was cooled in a water slide and pelletized. The composites were compounded in two steps. In the first step, PE was compounded with or without fire retardant. This was to ensure thorough mixing of the fire retardant in the PE. The melt temperature ranged from 192-201°C while the melt pressure ranged from 3.6-4.5 MPa. Prior to the second compounding step, WF was dried for 24 h at 105°C. The dried WF was then compounded with lubricant and PE with or without fire retardant as shown in Table 1. During the second compounding step the melt pressure ranged from 188-199°C while the melt pressure ranged from 4.2-7.0 MPa.

To form test samples for evaluating oxygen index, compounded pellets were dried at 105°C for at least 24 hours just prior to injection molding into flexural bar test samples. The composites were injection molded using a 33-ton Cincinnati Milacron (Batavia, Ohio) injection molder. The mold nozzle temperature was 188°C. The ASTM mold cavity used for the samples was 12.7 mm by 3.2 mm by 127 mm.

To form composite boards for cone calorimeter testing, the compounded pellets were dried at 105°C for at least 24 hours before being processed into boards using a Davis Standard 89-mm single-screw extruder (Pawcatuck, CT). The melt temperature ranged between 167-174°C. The extrudate was formed using a 12.7 mm x 127 mm (½” x 5”) radius-edge profile die.

Fire Performance Tests

We determined oxygen index according to ASTM D 2863 using procedure A [2]. The oxygen index value (OI) and corresponding estimated standard deviation were calculated according to the standard. Sample dimensions were as injection molded, a nominal 12.7 mm by 3.2 mm by 127 mm.

Cone calorimetry was performed on an Atlas Cone 2 Combustion Analysis System (Atlas Electrical Devices, Chicago, IL) according to ASTM E 1354 [3]. WPC samples were cut from the extruded boards to a size of 100 mm x 100 mm. The sample thickness was as extruded, at 12.7 mm. Samples were exposed in the horizontal orientation with the conical radiant electric heater located 25 mm above the specimen and the retainer frame (without the wire grid) over the test specimen. The sides and bottom of the samples were wrapped in aluminum foil. Each sample rested on an insulatory fiber blanket to keep it apart from the holder during the test. A spark igniter started the burning process and the length of time required to create a steady flame was recorded. Three replicate samples were tested at a heat flux level of 50 kW/m^2. The exhaust system flow rate was 0.024 m^3/s. For PE samples, data collection for the first replicate ended when it was visually observed that there was very little material in the foil wrapper. Due to a high heat release rate, data collection for the remaining two PE replicates was stopped early, but not before 300 seconds after specimen ignition. Data collection for WPCs was stopped once the mass loss rate dropped below 1.5 g/m^2-s. For reference, pine boards cut to 100 mm x 100 mm by 12.7 mm were also subjected to fire performance testing.

Ignitability was determined by observing the time for sustained ignition of the specimen, and is reported as ignition time (IT). The recorded initial peak HRR and calculated averages of the HRR over a set time (60 seconds and over the test duration) after ignition of the specimen was observed. The total heat release (THR), i.e., the cumulative heat release over the duration of the test was also calculated according to ASTM D 1354 [3].

Statistics

Each bar in Figure 4 and Figures 6-8 represents the mean of that data set and error bars represent one standard deviation. To determine significant differences between compared means, two-sample t tests were carried out at $\alpha = 0.05$. Tests for significance were two-tailed and assumed normal distribution and equal population variances. Letters above the bars in each of these figures denote significance. If the letters are the same, the hypothesis that the difference between means is zero was accepted. Conversely, different letters denote that the difference between means is not zero, i.e., the differences are statistically significant.

Results and Discussion

Fire Performance of Untreated WPCs

Oxygen index (OI) results for PE, WPCs, and solid pine are reported in Table 2. The OI of wood was higher than that of PE. This suggests that wood may have better flame retardancy characteristics than PE. The OI of WPCs was higher than PE, but lower than wood. Incorporating more wood into the composite (WF-60 versus WF-50) resulted in an improved OI.

Table 2. Oxygen Index results for polyethylene, 50% and 60% wood flour-polyethylene composites, and solid pine.

	Oxygen Index[a]
PE	19.3 (0.2)
WF-50	19.7 (0.1)
WF-60	20.2 (0.2)
Pine	23.9[b] (0.1)

[a] Reported values and estimated standard deviations in parentheses determined using ASTM D 2863 [16].
[b] Grain parallel to the long direction of the sample

Representative HRR versus time curves for PE and WPCs (WF-50 and WF-60) are shown in Figure 3. For comparison, a representative HRR curve for solid pine is included. Examination of the HRR versus time curve shows that the HRR of PE started increasing later than the HRR of pine. However, the HRR of PE continued to increase until the material was consumed, while the HRR of pine reached a peak early during the test duration, then reached a second peak later in the test. The peak HRR of PE was higher than the peak HRR for solid pine. The HRR of WPCs reached a peak early in the test, and then gradually decreased throughout the test. The peak HRR for WPCs fell between that of PE and solid pine.

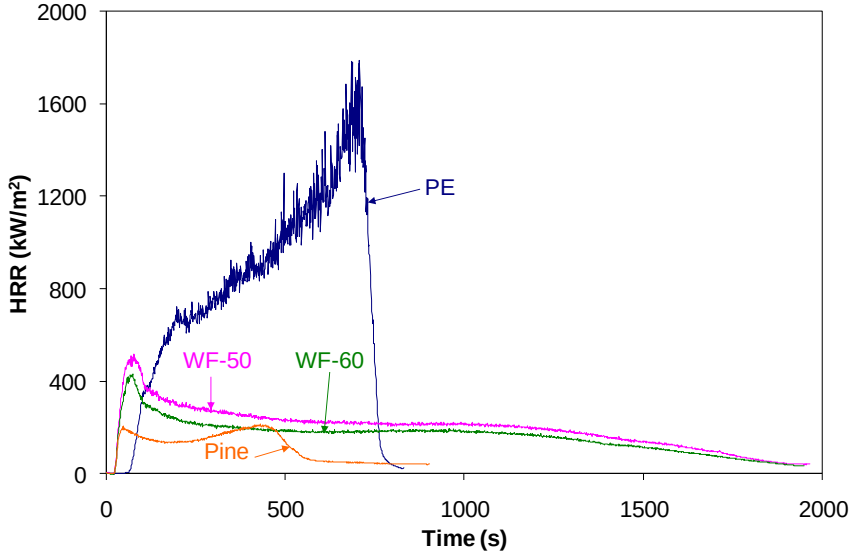

Figure 3. Representative HRR curves for polyethylene, 60% and 60% wood flour-polyethylene composites, and solid pine.

The cone calorimeter results for PE, WPCs (WF-50 and WF-60), and solid pine is shown in Table 3. A material performs best in a fire with a higher IT and lower HRR. Comparing the fire performance of PE with pine shows that although IT of PE is higher than pine, the calculated HRRs and THR are also higher for PE than pine. The results suggest that although it takes longer for PE to ignite compared with wood, once ignition is underway PE releases more heat and combustion consumes material faster than wood.

Table 3. Cone Calorimeter results for polyethylene, 50% and 60% wood flour-polyethylene composites, and solid pine.[a]

	IT[b]	Peak HRR[c]	Average HRR[c]		THR[d]
	(s)	(kW/m^2)	60 s (kW/m^2)	Total Test (kW/m^2)	(MJ/m^2)
PE	82.2 (9.5)	1790[e]	284 (21)	893[e]	609[e]
WF-50	24.5 (0.3)	505 (18)	369 (13)	207 (7)	373 (6)
WF-60	24.9 (0.3)	437 (15)	315 (3)	167 (7)	317 (10)
Pine	22.2 (2.1)	209[f] (2)	171 (11)	139 (7)	94 (6)

[a] Reported means with standard deviations in parentheses; [b] Ignition time (IT);
[c] Heat release rate (HRR); [d] Total heat release (THR); [e] Data for one sample only; [f] First peak

Generally, the fire performance based on cone calorimetry of WPCs fell between that of PE and solid wood (Table 3). The IT of WPCs was similar to IT wood and lower than PE. The peak HRR of WPCs was lower than PE and higher than solid wood. A comparison between the two WPCs showed that the WPC with more wood (60% versus 50%) had a lower peak HRR. This is consistent with an earlier study which reported a decrease in HRR with increasing wood content [5]. Borysiak et al. [6] also found that the wood-polypropylene composites had earlier

ignition times and lower peak HRR compared with the unfilled polypropylene samples. It is interesting that 60 seconds after ignition, the average HRR was higher for WPCs than for solid pine or PE. However for the duration of the test, the average HRR of WPCs was lower than the average HRR of PE and higher than the average HRR of pine. This was because WPCs reached their peak HRR during the first 60 seconds after ignition, while the HRR of PE increased throughout the test duration. The THR was also lower than PE, but higher than solid wood.

Both OI results and cone calorimetry results suggest that the fire performance of WPCs is better than PE but not as good as solid wood. This is consistent with better fire performance being obtained when PE is displaced with wood in a composite. For example, adding 60% wood by weight to PE decreased peak HRR of PE by 75% and THR by 48%.

Effect of Fire Retardants

Additive-type fire retardants were incorporated into WPCs to improve fire performance. We chose to compare results between WPCs with the same PE content. In our case, WF-60 and WPCs containing fire retardants all had a 35% PE content. In this manner incorporating fire retardants into the WPCs can be thought of as a partial replacement of wood with another filler.

OI and cone calorimetry were also used to evaluate the performance of fire retardants in WPCs. Each of the fire retardant systems had a positive effect on OI (Figure 4). Compared with WF-60, the most effective was AP, which increased OI by 29%.

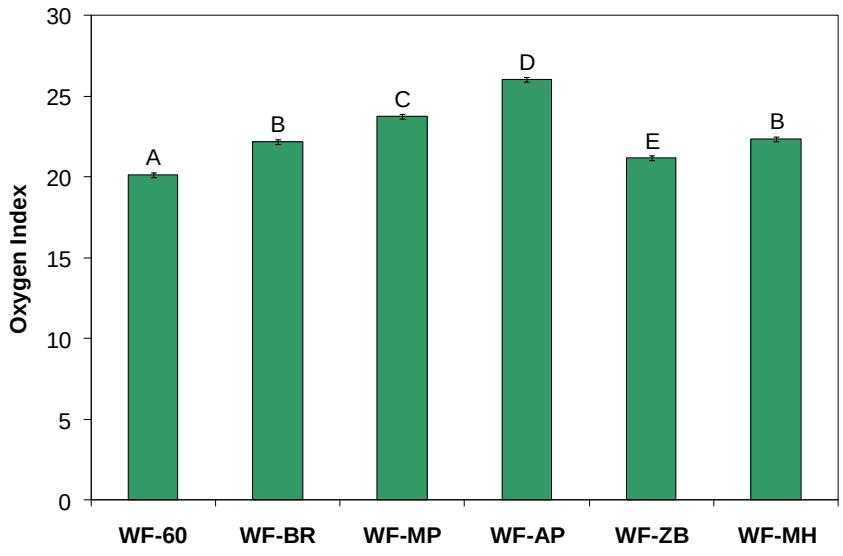

Figure 4. Oxygen index for 60% wood flour-polyethylene composites and for 50% wood flour-polyethylene composites with 10% fire retardant.

Representative HRR versus time curves for WPCs with and without fire retardants are shown in Figure 5. It is easily seen that all fire retardants lower HRR of WPCs. Overall AP decreased the HRR the most, but also led to a smaller HRR for a longer duration. Comparing

WPCs containing fire retardants, WPCs containing BR had the highest peak HRR while WPCs containing MH had the lowest.

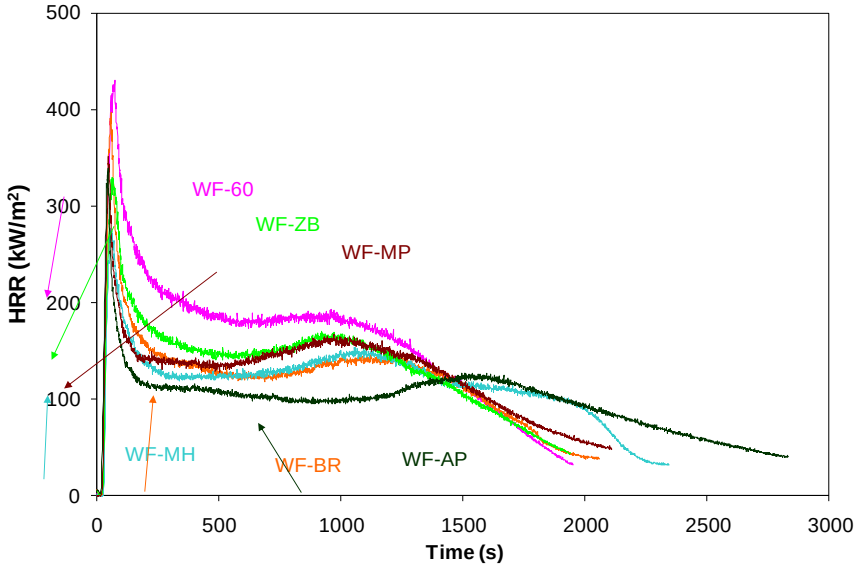

Figure 5. Representative HRR curves for 60% wood flour-polyethylene composites and for 50% wood flour-polyethylene composites with 10% fire retardant.

Incorporating fire retardants in WPCs had a mixed effect on ignition time (Figure 6). Fire retardants that significantly improved IT compared with WF-60 (i.e., increased it) include BR and MH. AP significantly decreased the ignition time. Compared with WF-60, MH increased IT by 24% and AP decreased IT by 12%.

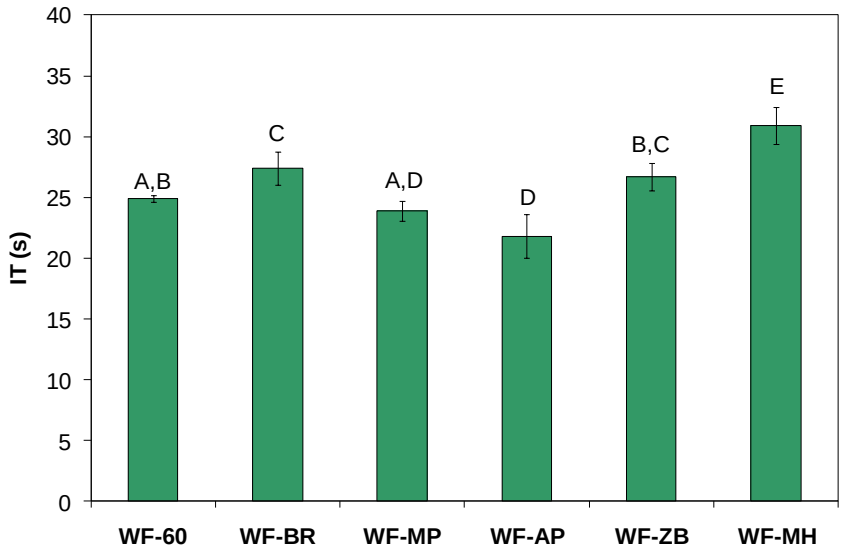

Figure 6. Observed ignition time (IT) for 60% wood flour-polyethylene composites and for 50% wood flour-polyethylene composites with 10% fire retardant.

Figure 7 summarizes the peak HRR as well as the HRR averaged over 60 seconds, 300 seconds, and the entire test duration once ignition was observed. All fire retardants significantly improved the peak HRR and average HRRs of WPCs. Compared with WF-60, the peak HRR of WPCs decreased between 11% and 35% when fire retardants were added. WPCs containing MH

performed the best while WPCs containing the BR performed the worst. Fire retardants also influenced the average HRR over the total test duration. Compared with WF-60, the average HRR over the test duration decreased between 19% and 39% when fire retardants were added. For average HRR, AP improved the fire performance of WPCs the most. Although ZB improved the fire performance of WPCs the least, the performance was not significantly different from MP and BR.

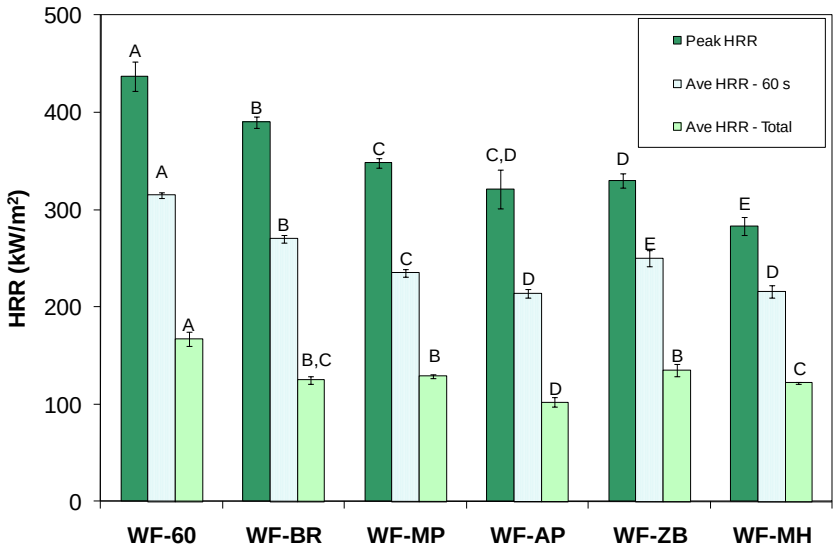

Figure 7. Calculated Heat Release Rates (HRRs) for 60% wood flour-polyethylene composites and for 50% wood flour-polyethylene composites with 10% fire retardant.

Each fire retardant system significantly lowered the THR, ranging between 15% and 24% improvement (Figure 8). Although BR improved THR the most, the performance was not statistically different from WPCs containing MP. While MH improved THR the least, the performance was not statistically different from WPCs containing AP.

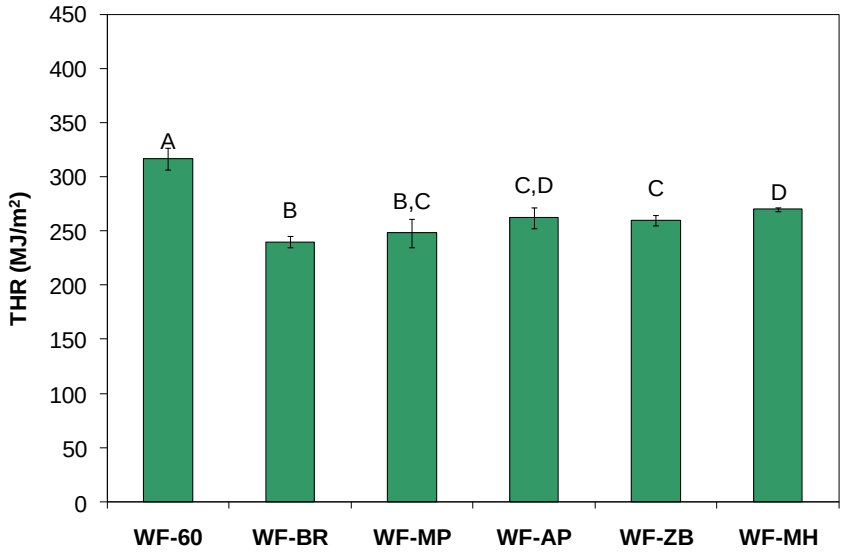

Figure 8. Calculated Total Heat Release (THR) for 60% wood flour-polyethylene composites and for 50% wood flour-polyethylene composites with 10% fire retardant.

Summary

There is potential for WPCs to expand into new and existing applications in the residential construction and furniture industries. However, incomplete knowledge regarding the fire performance of WPCs and the effectiveness of fire retardants limits this expansion. Typical lab-scale fire performance tests used for evaluating WPCs at the FPL include the oxygen index test and cone calorimetry. In the case study presented here, we used oxygen index tests and cone calorimetry to characterize the fire performance of wood flour-polyethylene composites compared with unfilled PE and solid wood. We also evaluated the effect of five additive-type fire retardants systems on fire performance.

The oxygen index of wood-polyethylene composites was reported to be higher than PE and lower than wood. The HRR of wood-polyethylene was generally lower than PE and higher than pine. The peak and average HRR for the test duration of WPCs was closer to wood than PE. This combination of results suggests that WPCs exhibit improved fire performance over PE, but perform worse than solid wood. Compared with PE, the ignition time of WPCs was shorter and 60 seconds after ignition the average HRR was higher. As the tests continued, the HRR of PE increased rapidly until the material was consumed, while the HRR of WPCs reached an initial peak and then decreased slowly. Therefore the fire performance of WPCs is better than unfilled PE, and worse than solid wood.

All fire retardant systems examined improved the OI and HRR of WPCs. Some fire retardant systems improved ignition time while others did not. The following summarizes the performance of the fire retardants studied.

- The bromine-based fire retardant generally performed the worst on the cone calorimeter tests. Improvements in peak HRR, average HRR after 60 seconds, average HRR for the test duration, MLR, and EHOC were all lowest for the bromine-based fire retardant.

- Magnesium hydroxide increased ignition time the most. During the initial stages of cone calorimetry, magnesium hydroxide performed well. The best improvements in peak HRR and average HRR after 60 seconds occurred with the addition of magnesium hydroxide. However, the THR was among the worst.

- Overall, zinc borate did not perform as well as the other fire retardants. The worst oxygen index was observed when zinc borate was used. At later times during cone calorimeter tests, the average HRR after 300 seconds and average HRR for the total test were the worst.

- Although melamine phosphate provided improved fire performance, it was generally neither the best nor worst performer in oxygen index tests and cone calorimeter tests.

- Ammonium polyphosphate generally performed well as a fire retardant. The best oxygen index, average HRR after 60 seconds, 300 seconds, and over the test duration, and mass loss rate were reported when ammonium polyphosphate was used. However, WPCs containing ammonium polyphosphate had the shortest ignition time, and the highest total heat release.

This study demonstrates that using oxygen index tests and cone calorimetry we can obtain a baseline of fire performance of WPCs and compare changes in fire performance when various additive-type fire retardants are incorporated into WPCs. FPL is continuing work in this area with several cooperators [7].

Acknowledgements

The authors gratefully acknowledge Scott Mueller and Tim Osswald (University of Wisconsin-Madison) as cooperators in the case study presented here.

All the cone calorimeter tests reported in this paper were conducted by Anne Fuller, cone calorimeter operator at the Forest Products Laboratory. Her contributions to the studies are gratefully acknowledged.

The authors also wish to thank American Wood Fibers, Struktol Company of America, Rio Tinto Materials, and Albemarle Corporation for the gracious donation of materials.

References

1. Clemons C. 2002. Wood-plastic composites in the United States: the interfacing of two industries. Forest Prod J 2002;52(6):10–18.

2. ASTM D 2863, Annual Book of ASTM standards. 8.01. West Conshohocken, PA: ASTM International.; 2008.

3. ASTM E 1354, Annual Book of ASTM standards. 4.07. West Conshohocken, PA: ASTM International; 2008.

4. Stark, N.M., White, R.H., Mueller, S.A., Osswald, T.A., 2010. Evaluation of various fire retardants for use in wood flour-polyethylene composites. Polymer Degradation and Stability. 95(9):1903-1910.

5. Stark N, White R, Clemons C. 1997. Heat release rate of wood plastic composites. SAMPE J 1997;33(5):26–31.

6. Borysiak S, Paukszta D, Helwig M. 2006. Flammability of wood-polypropylene composites, Polym Degrad Stab 2006;91(12):3339–3343.

7. White, R., Stark, N., Ayrilmis, N., 2011. Recent Activities in Flame Retardancy of Wood-Plastic Composites. In: Proceedings of the 21st BCC Conference on Recent Advances in Flame Retardancy of Polymeric Materials, May 23-25, 2011, Stamford, CT.

In: Proceedings of the 5th International Wood Fibre Polymer Composites Symposium. 2011 September 26-27. Biarritz, France. FCBA Institut Technologique: Paris, France. 12 p.