TEN DECADES OF FLAME RETARDANCY TESTING AT THE U.S. FOREST PRODUCTS LABORATORY

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ABSTRACT

The development or adoption of advances in flammability test equipment and methods played a critical role in facilitating ten decades of FPL research to reduce the flammability of wood products. FPL equipment developments include the 1912-1915 work of R.E. Prince to expose specimens to constant temperatures to determine the times for ignition, the fire tube apparatus in 1920's, and the 8-ft tunnel in 1950's. In the 1960's, considerable fundamental research was conducted using thermogravimetric and differential thermal analysis. As an early promoter of the use of heat release rate as a measure of relative flammability, FPL developed an apparatus in the 1960's to measure the heat released by a burning material. As better technologies were developed to measure heat release rate, FPL obtained such equipment to continue its efforts to properly measure the potential contribution of wood products to a fire and facilitate the development of new fire-retardant-treated wood products.

INTRODUCTION

The U.S. Department of Agriculture-Forest Service Forest Products Laboratory (FPL) was established on the campus of the University of Wisconsin (Madison, WI) in 1910. From its early days, research on fire-retardant (FR) treatments has been part of its research activities. A key component of the FR research efforts was the development and use of fire test methodologies to evaluate improvements in performance. Measurements of improved fire performance include times for ignition, mass loss, visual observations, temperature and heat release rate.

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TIMES FOR IGNITION

In the 1910's effort of R.E. Prince (1), times for ignition were used to evaluate fire-retardant-treated (FRT) wood. The wood specimens were exposed to constant temperature conditions in a quartz cylinder with electric heating elements. He also developed a second apparatus with a silicate plate heated to 325°C using flat nichrome wire to exposed the test specimen in the presence of a gas pilot flame (1, 2) to test larger samples of commercial products. In current ignition research, the fire exposure is often defined in terms of a constant heat flux. Ignition was not considered an effective parameter to evaluate FR treatments. Recent ignition research is focused on data for fire models (3).

MASS LOSS

In some tests of Prince, the weight loss of the specimens was also recorded (2). When exposed to heat, wood undergoes thermal degradation to volatiles gases and char. Many FR treatments change the thermal degradation process so less wood degrades to the volatile gases and more residual char is left. This phenomenon has been used to evaluate the effectiveness of FR treatments. In particular, it is the basis for the fire tube test and thermal gravimetric analysis. Measurement of mass loss is part of smoldering tests such as the tests for cellulosic insulation (4, 5).

Fire Tube Test

In the 1920's, M.E. Dunlap initiated development of the fire tube apparatus (6). A sample is inserted in a metal tube hanging from a scale and a burner is inserted beneath the sample. Progressive weight loss of the sample and the temperature at the top of the tube are recorded. An effective FR treatment results in high residual mass due to extinguishment of flames when burner is removed and higher residual char. The method became ASTM standard E 69 in 1946.

With the fire tube apparatus, T.R. Truax and C.A. Harrison investigated about 130 treatments of inorganic chemicals in the early 1930's (7). The research concluded that combinations of chemicals were the best way to obtain effective FR treatments for both flaming and glowing. These salts were extensively used in the first generation of commercial FRT formulations. We continue to use the fire tube tests for some studies (8-10). Another similar test is the crib test in which a small crib is ignited with a burner and weight loss is recorded (11). This test method evolved into ASTM E 160 which was approved in 1941 as C 160 and withdrawn as an ASTM standard in 1993.

Thermal Analysis

At the time of the FPL 50th Anniversary in 1960, the fire retardancy program shifted to fundamental research on thermal degradation and combustion of wood (12, 13). Browne's 1958 report (14) provided an extensive review of the theories of "flameproofing", chemical mechanisms of retardants, and theories of glow prevention. In thermogravimetric analysis (TGA), the mass loss of a very small sample exposed to elevated temperatures in either air or nitrogen environments is recorded. In other thermal analysis techniques such as differential calorimetric analysis, temperature measurements are made to identify and measure the heat of reactions of the thermal degradation. Considerable

fundamental research was conducted by F.L. Browne (15-17), W.K. Tang (18, 19), J.J. Brenden (20), and F.C. Beall (21, 22) during this time period. TGA and other thermal analysis techniques continue to be used in research on new FR treatments (23-25).

VISUAL FLAME SPREAD

One obvious way to evaluate a FR treatment is to subject the sample to heat source or flames and make visual observations of the flame height or the spread of flames over the surface of the specimen. Such tests include the 8-foot tunnel, FPL modified Schlyter test, and full-scale tests.

8-Ft Tunnel

In the 1950's, T.R. Truax, H.D. Bruce and V.P. Miniutti developed the 8-ft tunnel test in which observations are made of the flame spread along the length of the specimen (26-28). A burner at one end heated a metal plate that provided the external heat exposure. It was modeled in part after the regulatory ASTM E 84 tunnel test (11). The method was standardized as ASTM E 286 in 1965 and withdrawn as a standard in 1991. In tests of eleven materials, good agreements were obtained between the 8-ft tunnel and the FPL corner-wall test (11). With the development of the 8-foot tunnel, FPL continued to investigate FR formulations including organic and inorganic chemicals (12) in the 1960's and 1970's (29). H.W. Eickner and E.L. Schaffer evaluated eleven treatments for plywood using the fire tube, FPL Schlyter panel test, and the 8-foot tunnel test (30). Increasingly, the focus of the research was on FR treatments for composite products (31, 32). The exploratory study by A.D Syska involved 15 potential treatments for particleboard (31). Many of the projects on FR treatments were cooperative efforts with non-FPL researchers. Research with the 8-foot tunnel included studies examining wood-base standard reference materials (33, 34). While the 8-ft tunnel test was used for many years for FR research, it was limited in its ability to identify highly effective treatments or highly flammable materials in a manner consistent with ASTM E 84.

Other Laboratory Tests

Over the years, FPL has examined a wide range of flammability test equipment. A 1959 publication on FPL fire test methods (11) listed the fire tube, modified Schlyter, 8-foot tunnel, crib test, inclined panel test, roof-corner test, sidewall test, and the SS-A-118a fire test as spread of flames tests. In the FPL adaptation in the 1930's of a test developed by R. Schlyter of Sweden (35), a burner is placed between two vertical pieces of the test material. Visual observations are made of the heights of the flames. It was found useful for studies on FR coatings and FRT wood shingles (8). After a series of fiber board tests in the 1950's using the SS-A-118a panel test, FPL modified Schlyter test and British Standard Specification No.476 inclined-panel test, Van Kleeck and Martin (36) noted the difficulty of consistently predicting performance in one test using the results of another test method.

T.H. Wegner and C.A. Holmes used the critical radiant panel test apparatus (ASTM E 970/ E 648) in the 1970's to study boron treatments for cellulosic insulation (4). C.A. Holmes examined the flammability of wood products in the test of Motor Vehicle Safety Standard No. 302 (36a). R.H. White examined the use of the oxygen index test (37) for wood. M.A. Dietenberger has used the Lateral Ignition Flame Test (LIFT) test (ASTM E 1317/E 1321) to obtain flame spread and ignitability data for fire modeling (38).

Full-scale Tests

Full-scale tests have an early beginning in FPL fire research. In the late 1940's, a series of "dwelling" tests included furniture, other "contents" and wall linings in a 2.4 m by 3.6 m foot room (39). The effect of wall linings was also investigated in corridors of an abandon school building (40). FPL test methods in a 1959 publication (11) included a roof-corner test, sidewall test, and corner wall test. During WW II, tests were conducted in stimulated attic spaces to investigate thick coatings as protection from incendiary-type bombs (41). The first corner-wall test in an enclosed room to be conducted at FPL occurred in 1949 (42). The specimen consisted of a corner of 0.6 m wide panels on two walls and sections on ceiling. Initially alcohol was used as the ignition but later wood cribs were used. Corner-wall tests included tests on plywood (43), fiber insulation board (36) and plywood-faced foam plastic and paper honeycomb sandwich panels (42). The standard ASTM E 2257 room test is now used by FPL for such tests (44). Three walls, and in some cases the ceiling, of the room are lined with the test material and a propane burner is placed in one corner of the room. In addition to heat release measurements using oxygen depletion techniques, visual observations are made of the times for flashover. A joint U.S.-Slovak series of tests (45) showed that the 100 kW/ 300 kW burner profile and test material only on the walls produced relative times for flashover for various wood products consistent with the rankings in the ASTM E 84 tunnel test.

HEAT RELEASE RATE

FPL was an early promoter of rate of heat release (RHR) as a measure of relative flammability. It was realized that RHR would provide a better indication of the contribution of wood to a fire than total heat content from the oxygen bomb test. J.J. Brenden developed the FPL RHR apparatus in the 1960's to measure the heat released by a burning material (46-48). Initially, material-energy balance approach was used but later a substitution method was used to obtain RHR. As better technologies were developed to measure RHR, FPL obtained such equipment to continue its fire modeling research and facilitate the development of new FRT wood products.

In the 1980's, the original FPL RHR apparatus was replaced with an Ohio State University (OSU) RHR apparatus (ASTM E 906). The method uses thermopiles to measure the heat released by the burning samples. The OSU RHR apparatus became the methodology of choice for FPL FRT research activities (8, 9). In addition to the RHR, the apparatus provided a measure of the times for ignition. H.C. Tran modified the FPL OSU RHR apparatus to measure the HRR using the oxygen depletion technique (49, 50). He also compared results from the O.S.U. apparatus and the cone calorimeter (51). The thermopile technique is used to provide a measure of the heat release rate in the mass loss calorimeter (ASTM E 2102), a screening tool for FR research.

In the 1990's, the OSU RHR at FPL was replaced with a cone calorimeter apparatus. In the cone calorimeter (ASTM E 1354), oxygen consumption is used to measure the RHR (52). Like the OSU apparatus, the cone calorimeter became the primary fire test for FPL FRT research (53-55). In addition to HRR and times for ignition, the cone calorimeter records the mass loss and an effective heat of combustion. The emphasis of the FR research continued to be on FR treatments for composite materials (56, 57). Like the OSU apparatus, FPL used its cone calorimeter to develop fundamental property data for both untreated and FR treated wood products that can be used in fire growth models (52, 58). The development by M.A. Dietenberger of models to predict the ASTM E 84 flame spread index and flame spread in the room corner test (59, 60) improved the utility of the cone calorimeter for FR research. FPL is posting on its web site the data files for cone calorimeter tests cited in its publications (http://www.fpl.fs.fed.us/products/products/cone/introduction.php).

SMOKE DEVELOPMENT

In addition to flammability, smoke and toxic gases contribute to reduced fire safety in a building. While FPL has not conducted research on gas toxicity, it did work on methods for measuring obscuration due to smoke. Early work in 1960's and 1970's concentrated on light-transmission properties of smoke from radiant heated untreated and FRT wood (61, 62). The National Bureau of Standards (NBS) Smoke Chamber was adopted as ASTM standard E 662 in 1979. Considerable work was done at FPL with the NBS chamber (63-66). The OSU RHR apparatus (67), cone calorimeter and the room tests are tests in which dynamic measurements are made of the visible smoke. Visible smoke measurements are also made in the 8-ft tunnel and 25-ft tunnel tests. FPL efforts have included developing correlations between the small-scale tests and the full-scale room test (68) and the different small-scale tests (66).

OTHER PERFORMANCE CHARACTERISTICS

In addition to improving the fire performance, a FR treatment must have other performance characteristics (69). The need to examine effects of fire retardants on adhesive quality, strength, and corrosion was realized in early FPL research on FRT wood. Concerns about non-fire performance requirements have driven improvements in FR treatments. Interactions of FR chemicals with adhesives (70-72), effects of FR treatments on charring rate (73), resistance of FRT wood to decay (74, 75) have been investigated.

FPL researchers starting with Prince in 1915 (76) have been concerned about FR chemicals increasing corrosion of metal connections. In the 1940's, a technique was developed to use metal test coupons to evaluate the corrosion potential of FR solutions (76). The development of the second generation of treatments in the 1970's was driven by concerns about corrosion and the need for treatments with lower hygroscopicity (77). ASTM standard for hygroscopity of FRT wood is D 3201. With the replacement of CCA with new commercial preservative treatments for wood, the effect of chemical treatments on corrosion is again an active area of FPL research (78, 79).

In exterior applications such as wood shingles, exposure to rain and UV must not adversely affect the effectiveness of the FR treatment. In the late 1960's and early 1970's, research by C.A. Holmes on accelerated weathering of wood shingles prior to fire testing resulted in the FPL weathering chamber (80-83). In addition to another protocol used by Underwriters Laboratories, the FPL test protocol was incorporated in ASTM D 2898. The research in this area included the evaluation of treated wood shingles after 10 years of actual outdoor exposure (84, 85).

Before any structural wood product is exposed to a fire, it will be subjected to structural loads. Thus, the structural design values must be adjusted for the impact the FR treatment has on the mechanical properties (86). Early work on treatments for wood included the evaluation of their effects on strength properties (86, 87). Works on fire retardant treatments for composite products often include the evaluation of their effects on strength properties (88).

In the 1980's, there were reports of FRT plywood roof sheathing degrading and failing structurally (89). The subsequent development of the "third generation" of FRT formulations was the result of these structural failures of some first and second generations of FRT wood products. A major FPL research effort by J.E. Winandy, S.L. LeVan, R.J. Ross, and others provided the understanding needed to address the problem and identify treatments that were susceptible to failure when exposed to elevated temperatures (90-92). Test protocols were developed (93) and standardized in ASTM International for plywood (D 5516, D 6305) and lumber (D 5664 and D 6841). Reports on such testing include ones for a boron-nitrogen, phosphate-free fire retardant treatment (94-96) and a GUP-B treatment (97).

CONCLUDING REMARKS

In this paper, we have reviewed ten decades of fire retardancy research at the USDA, Forest Service Forest Products Laboratory in the context of the test methods used to evaluate the properties of the FRT wood products. Previous reviews of FPL's fire research include a 1954 presentation by Truax (98) and one by Eickner (12) written for the 50th Anniversary of FPL. FPL articles on FRT wood include those by Truax (99), Van Kleeck (100), Eickner (101), Holmes (102), LeVan (103) and Sweet (104). As we begin the 11th decade of fire research, FPL is constructing a new building for fire research. The Centennial celebration of the Forest Products Laboratory will include the publication of a Centennial edition of its *Wood Handbook* which has a chapter on fire safety of wood products. Future FR research at FPL will likely continue to emphasize the treatment of composite materials including wood-plastic composites (54, 105, 106).

We thank BCC Research and Professor Menachem Lewin for providing these annual conferences on recent advances in flame retardancy of polymeric materials as an outlet for our research results for the past two decades (25, 54, 56-60, 83, 104, 107, 108).

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