

Wood Products: Thermal Degradation and Fire[☆]

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1 Thermal Degradation

As wood reaches elevated temperatures, the different chemical components undergo thermal degradation that affect the performance of wood. The extent of these changes depends on the temperature level and length of time under exposure conditions. Permanent reductions in strength and modulus of elasticity can occur at temperatures > 65 °C, with the amount depending on the temperature, pH of wood, moisture content, heating medium, exposure period, chemical treatment, and species (Winandy and Rowell, 2013). Studies relating strength reduction with temperature rise has indicated a kinetic basis (involving activation energy, pre-exponential factor, and order of reaction). If the specimens are tested while still hot after long term exposure (~ 66 °C for 3 years), the percentage of strength reduction would be based on the combination of an immediate effect of temperature ($\sim 14\%$) and a permanent effect ($\sim 33\%$) for a total of around 47% strength loss (Green and Evans, 2008). The primary mechanism for this degradation is the production of acetic acid freed by the process of hydrolysis at long term ambient conditions that include varying moisture contents and by which the acetyl groups are split from their association with hemicelluloses in the wood (Packman, 1960). During hydrothermal treatments (as reported for up to 230 °C), the acetyl groups present in wood hemicelluloses hydrolyze to form acetic acid which provides the hydronium ions needed to self-catalyze the hydrolysis of wood, involving the hydrolytic reactions of ether and ester bonds (Garrote *et al.*, 2002). Long term exposure to temperatures equal to or greater than 66 °C is not a concern in residential construction but may be an important consideration for certain industrial buildings. However, strength and stiffness degradation relationships with temperatures greater than 66 °C and varying time durations for wood under compression and tension has recently been utilized in building design for fire protection (Eurocode 5, 2004).

Auto-oxidation in wood can proceed, although imperceptibly at low temperatures, after the oxidizing aldehydes and other radicals become more available from auto-hydrolysis, although fatty acids naturally in the wood has also been postulated for auto-oxidation. Long term storage of wood chips or pellets utilized in home heating has been known to develop potentially lethal levels of CO, postulated to be caused by auto-oxidation, unless adequate ventilation is provided (Gauthier *et al.*, 2012).

1.1 Temperature Regimes of Wood Mass Loss

Various chemical bonds begin to break at temperatures > 100 °C that are manifested as extractive, carbohydrate and lignin weight losses of various types that increase with the temperature (wood pyrolysis). Literature reviews on thermal degradation can be found in Bryden (1998), Müller and Bellan (1996), Shafizadeh (1984), Atreya (1983), Beall and Eickner (1970), and Browne (1958). These reviews indicate that thermal degradation above 100 °C can be broken up into four temperature regimes:

- (i) Between 100 and 200 °C, wood becomes dehydrated, generating water vapor and other noncombustible gases and liquids including CO₂, CO, formic acid, acetic acid, and glyoxal. Auto-hydrolysis and auto-oxidation are chemical mechanisms that become more pronounced. At 130 °C, volatile organic compounds (VOC) can be detected, and with water vapor and CO₂ from dehydration and decarboxylation processes, the carbohydrate polymers reduce very slowly to a mostly carbon residue, char. With a slow heating rate of 0.3 °C min⁻¹, natural extractives decompose between 150 and 200 °C.

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Exothermic oxidation reactions can occur because ambient air can diffuse into, and react with, the developing porous char residue.

- (ii) From 200 to 300 °C, some wood components begin to undergo significant pyrolysis, and in addition to the gases and liquids listed previously, significant amounts of CO and high-boiling-point tar are given off. The hemicelluloses and lignin components are pyrolyzed in the ranges 200–300 °C and 225–450 °C, respectively. Much of the acetic acid liberated from wood pyrolysis is attributed to deacetylation of hemicellulose. Dehydration reactions accelerating around 200 °C are primarily responsible for pyrolysis of hemicellulose and lignin and result in a high char yield for wood. Although cellulose remains mostly unpyrolyzed, its thermal degradation can be accelerated in the presence of water, acids, and oxygen. As the temperature increases, the degree of polymerization of cellulose decreases further, free radicals appear, and carbonyl, carboxyl, and hydroperoxide groups are formed. Overall, pyrolysis reactions are endothermic as consistent also with decreasing dehydration and increasing CO formation from porous char reactions with H₂O and CO₂ with increasing temperature. During this 'low-temperature pathway' of pyrolysis, exothermic reactions of exposed char and volatiles with atmospheric oxygen are manifested as glowing combustion.
- (iii) The third temperature regime is from 300 to 450 °C because of the vigorous production of flammable volatiles. Between 300–350 °C, significant depolymerization, which is highly endothermic, of cellulose occurs instead of the char forming dehydration reactions. At high enough temperature rise rate, char forming dehydrations can effectively be bypassed, creating high amounts of tar. At high pressures and high residence time, the cellulosic tar will degrade into secondary char and off-gases in greater amounts. Also, around 300 °C aliphatic side chains start splitting off from aromatic rings in the lignin. Finally, the carbon–carbon linkage between lignin structural units split around 370 to 400 °C. The degradation reaction of lignin is an exothermic reaction, with peaks occurring between 225 and 450 °C. The temperatures and amplitudes of these peaks depend on whether samples were pyrolyzed under nitrogen or air. All wood components end their volatile emissions at around 450 °C. The presence of minerals and moisture within the wood tends to cause the separate pyrolysis processes of the major wood components to overlap and interact. In this 'high-temperature pathway,' pyrolysis of wood results in overall low char residues of around 25% or less of original dry weight. Many fire retardants work by shifting wood degradation to the 'low-temperature pathway,' which reduces volatiles available for flaming combustion.
- (iv) At >450 °C, the remaining wood residue is a non-volatizing char, which undergoes further degradation by being oxidized to CO₂, CO, and H₂O. This is referred to as afterglow.

1.2 Kinetic Parameters of Wood Pyrolysis

The complex nature of wood pyrolysis often leads to the selection of empirical kinetic parameters of wood pyrolysis applicable to specific cases. For wood degrading at relatively low temperatures for a long period and ignoring volatile components, a simple first-order reaction following the Arrhenius equation was found practical (Stamm, 1955):

$$\frac{dm}{dt} = -A \exp\left[-\frac{E}{RT}\right] \quad [1]$$

where m is the mass of the specimen, t is time, A is the pre-exponential factor, E is the activation energy, R is the universal gas constant, and T is the temperature in Kelvin. The simplest heating environment for determination of these kinetic parameters is isothermal, constant pressure, and uniform flow gas exposures on a nominally thick specimen. As an example, Stamm reported mass loss of coniferous wood sticks (25 by 25 by 150 mm; southern and white pine, Sitka spruce, and Douglas fir) that were heated in a drying oven over a temperature range of 93–250 °C. The fit of Arrhenius equation to data resulted in values of $A = 6.23 \times 10^7 \text{ s}^{-1}$ and $E = 124 \text{ kJ mol}^{-1}$. When these wood sticks were exposed to steam instead of being oven dried, degradation was much faster, with corresponding kinetic parameters of $A = 82.9 \text{ s}^{-1}$ and $E = 66 \text{ kJ mol}^{-1}$. Stamm concluded that steam acted as a catalyst because of significant reduction in the value of the activation energy. Shafizadeh (1984) showed that pyrolysis proceeds faster in air than in an inert atmosphere and that this difference gradually diminishes around 310 °C. Values of activation energy reported for pyrolysis in air varies from 96 to 147 kJ mol⁻¹.

In another special case, a simple dual reaction model could distinguish between the low- and high-temperature pathways for quantifying the effect of fire retardant on wood pyrolysis (Tang, 1967):

$$\frac{dm}{dt} = (m_{\text{end}} - m) \times \left[A_1 \exp\left(-\frac{E_1}{RT}\right) + A_2 \exp\left(-\frac{E_2}{RT}\right) \right] \quad [2]$$

In this equation, m_{end} is the ending char mass, and subscripts 1 and 2 represent low- and high-temperature pathways, respectively. Dynamic thermogravimetry was used to span the temperature to 500 °C at a rate of 3 °C min⁻¹ using very small wood particles of ponderosa pine sapwood, lignin, or α -cellulose samples with five different inorganic salt treatments as shown in Figure 1. Tang's derived values for the untreated wood were $m_{\text{end}} = 0.21$ of initial weight, $A_1 = 3.2 \times 10^5 \text{ s}^{-1}$, $E_1 = 96 \text{ kJ mol}^{-1}$, $A_2 = 6.5 \times 10^{16} \text{ s}^{-1}$, and $E_2 = 226 \text{ kJ mol}^{-1}$.

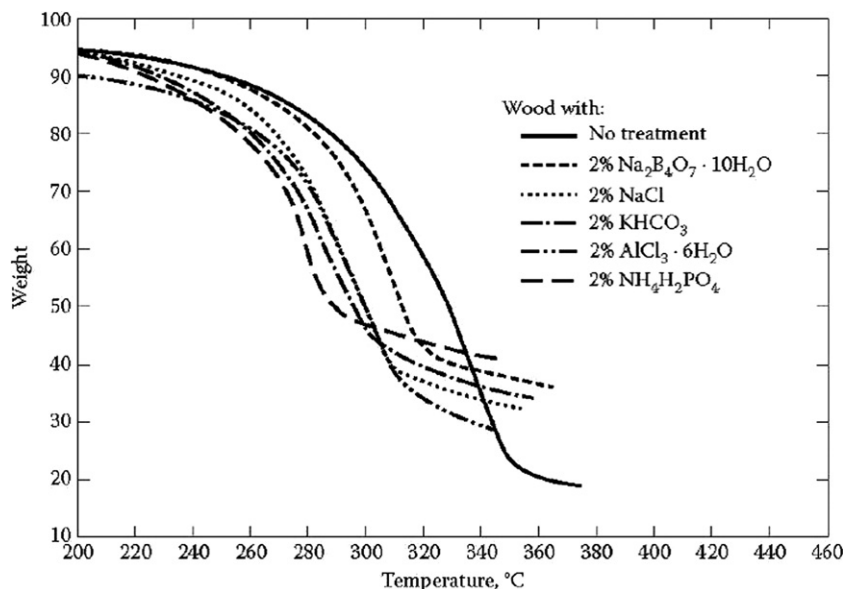


Figure 1 Thermogravimetric analysis of wood treated with various inorganic additives. (Adapted from Tang, W.K. and Eickner, H.W., 1967.)

A well-known flame-retardant-treatment (FRT) chemical, monoammonium phosphate, was the most effective tested in that char yield was increased to 40% and E_1 decreased to 80 kJ mol^{-1} , thereby promoting volatile loss through the low-temperature pathway. α -Cellulose reacted to the chemicals in a similar manner to the wood, and lignin did not seem to be affected much by the chemicals. Thus, these FRT chemicals significantly reduced the flammable volatiles generated by the cellulose component of wood. Potential FRT chemicals are often initially tested using dynamic thermogravimetry or differential scanning calorimetry. However, the FRT chemical monoammonium phosphate has also been found to cause the greatest strength loss of wood exposed to elevated temperatures $> 65 \text{ }^\circ\text{C}$ (Winandy and Rowell, 2013), leading one to consider more careful FRT selection.

For applications to biomass energy and fire growth phenomenology, kinetic parameters become essential to describe flammable volatiles and their heat of combustion, but are very complicated. Using a sophisticated pyrolysis model, Bryden (1998) showed that to maintain a uniform temperature profile at very high temperatures under isothermal or uniform rate of temperature rise conditions, the wood particles are required to be less than a fraction of a millimeter. In thermogravimetric analysis, this small size factor is not a difficulty, except that the pyrolysis is no longer realistic. This is because in real applications: (1) evaporating moisture creates high pressures resulting in Darcy flow of steam and volatiles in the thick porous wood, (2) tar decomposes when passing through porous char, and (3) heating rate is both locally high and low so that all four regimes of wood pyrolysis are involved in a single wood slab.

Pyrolysis models such as those of Dietenberger (2012), Bryden (1998), and Miller and Bellan (1996) include competing reactions to produce char, tar, and noncondensing gases from wood as well as the secondary reaction of tar decomposition. Bryden's model incorporates reactions of CO_2 , CO , and H_2O with char to form CO gas that converts to CO_2 during glow combustion. Miller and Bellan's model uniquely obtains 'robust' pyrolysis of the wood components, hemicellulose, lignin, and cellulose, such that various oven-dried wood species can be accommodated without changing the constants. Dietenberger's model obtains pyrolysis of extractives, holocellulose, and lignin that include competing reactions between dehydration and devolatilization and with the char glowing reactions while calculating volatile's heat of combustion, carbon content, and evolving mass rates as a function of material's dynamic temperature. Uses of such sophisticated models are limited by their numerous empirical kinetic and thermophysical parameters, certain phenomenology incompleteness, and large computation times.

2 Fire Performance Properties of Wood

In building applications, fire performance of wood and other combustible materials are regulated based on performance in standard tests. Although improvements have been made in standard tests for fire performance, results of such tests are only applicable for fire exposure and other conditions of the standard test. In this section, the fire performance of wood is reviewed in the context of general fire performance and regulatory tests (White and Dietenberger, 2010).

2.1 Ignition

Ignition of wood is the start of visual and sustained combustion (smoldering, glow, or flame) fueled by wood pyrolysis. Therefore, the flow of energy or heat flux from a fire or other heated object to induce pyrolysis is a necessary condition of ignition. Sufficient

conditions of flaming ignition are the mixing together of combustible volatiles and air in the right composition and a temperature from 400 to 500 °C. An ignition source (pilot flame or spark plug) is therefore usually placed where optimum mixing of volatiles and air can occur for a given ignition test. In such tests, the surface temperatures of wood materials have been measured in the range 300–400 °C prior to piloted ignition. This coincides with the third regime of wood pyrolysis in which there is a significant production of flammable volatiles. However, it is possible for smoldering or glowing to exist prior to flaming ignition if the imposed heating causes the wood surface to reach 200 °C or higher for the second regime of wood pyrolysis. From tests that imposed a constant external heat flux to the wood surface, values for ignition temperature, minimum flux needed for ignition, and thermophysical properties have been derived. Density and moisture content have a large influence on these material-dependent properties. Critical heat flux for ignition has been calculated to be between 10 and 13 kW m⁻² for a range of wood products. For exposure to a constant heat flux, ignition times for solid wood typically ranged from 3 s for heat flux of 55 kW m⁻² to 930 s for heat flux of 18 kW m⁻².

Unpiloted ignition is flaming ignition that occurs in the absence of a pilot source. The external heat flux needed for unpiloted ignition is around 25 kW m⁻², depending on the apparatus. For radiant heating of cellulosic solids, unpiloted transient ignition has been reported at 600 °C. With convective heating, unpiloted ignition of wood has been reported as low as 270 °C and as high as 470 °C. Unpiloted ignition that involves wood exposed to low level external heat sources for very long periods is an area of dispute. Schaffer (1980) used a residual weight criterion of 40% of the initial weight to suggest that wood can safely be heated to 150 °C for a year or more. This temperature criterion is based on the assumption that thermal degradation is a necessary condition for such ignition. Evidence that such a low temperature criterion is a sufficient condition for ignition has not been successfully documented. Temperatures as low as 70 °C are recommended for wood exposed for long periods to prevent significant permanent loss of properties as well as prevent ignition.

A special type of unpiloted ignition is spontaneous ignition that occurs when heat generated from an internal source is not readily dissipated. Such ignition generally occurs for a prolonged period and involves smoldering. Continuous smoking is visual evidence of smoldering, which is sustained combustion within the pyrolyzing material. Although smoldering can be initiated by an external ignition source, a particularly dangerous smoldering is that initiated by internal heat generation. In order of increasing temperature, potential mechanisms include respiration, metabolism of microorganisms, adsorptive heat, heat of pyrolysis, and abiotic oxidation (Kubler, 1990). If such internal heat generation is sustained by a continuous supply of fresh air and sufficiently contained by the material's thermal insulation, internal temperatures will continuously increase, leading first to smoldering and sometimes further to flaming ignition. Examples of such fires include panels or paper removed from a press or dryer and stacked in large piles without adequate cooling and very large piles of chips or sawdust subjected to exothermic biological activities.

2.2 Rate of Heat Release

Heat of combustion measured in an oxygen bomb calorimeter is the total heat available. Higher heating values for wood are about 20 kJ kg⁻¹, which includes heat of condensation of water vapor produced from combustion of fuel. Heat of combustion depends on the relative lignin and holocellulose and extractive contents of wood. Cellulose and hemicellulose have a higher heating value of 18.6 kJ kg⁻¹, whereas lignin has a higher heating value of 23.2–25.6 kJ kg⁻¹. Higher heating values of extractives are about 32–37 kJ kg⁻¹.

In a fire situation, the contribution of combustible materials to a fire depends more on the heat release rate (HRR) rather than the total heating value. The best known method for determining HRR is the American Society of Testing Materials (ASTM) E1354 (also ISO 5660), known as the cone calorimeter which is based on the oxygen consumption method (Babraukas and Grayson, 1992). With untreated wood, as shown in Figure 2, the HRR increases to a peak shortly after ignition, then decreases to a lower semi-constant HRR when exposed to a constant heat flux. The char layer provides thermal insulation from the fire and gradually reduces the rate of charring propagation, thus also the HRR. Wood specimens tested with an insulating backing will also have a second peak in HRR due to termination of the thermal wave and the afterglow phenomena. By increasing the imposed heat flux, the ignition and burn time is shortened, and the peak HRR is increased. In general, the averaged effective heat of combustion in cone calorimeter tests of wood is about 65% of the higher heating value from an oxygen bomb.

2.3 Charring and Fire Resistance

When wood is exposed to high temperatures it develops an insulating layer of char that retards further degradation of the wood (White and Diitenberger, 2010). The load-carrying capacity of a structural wood member depends upon its cross-sectional dimensions as shown in Figure 3. Thus, the amount the cross section charred is a major factor in the fire resistance of structural wood members. The depth of the char formed over time is called the charring rate. In the standard ASTM E 119 fire-test exposure, ignition occurs in about 2 min and charring into the depth of the wood then proceeds at a rate of approximately 0.8 mm min⁻¹ for the next 8 min (or 1.25 min min⁻¹). Once enough char is formed, it has an insulating effect and the rate decreases to 0.6 mm min⁻¹ (1.6 min min⁻¹). Considering the initial ignition delay, the fast initial charring, and the insulating effect of char, the average constant charring rate is about 0.6 mm min⁻¹ (or 1.5 in h⁻¹) (Douglas-fir, 7% moisture content).

Material properties that vary among species such as density, chemical composition, and permeability, as well as the moisture content, will affect the charring rate. Density relates to the mass subjected to degradation and of the corresponding thermal

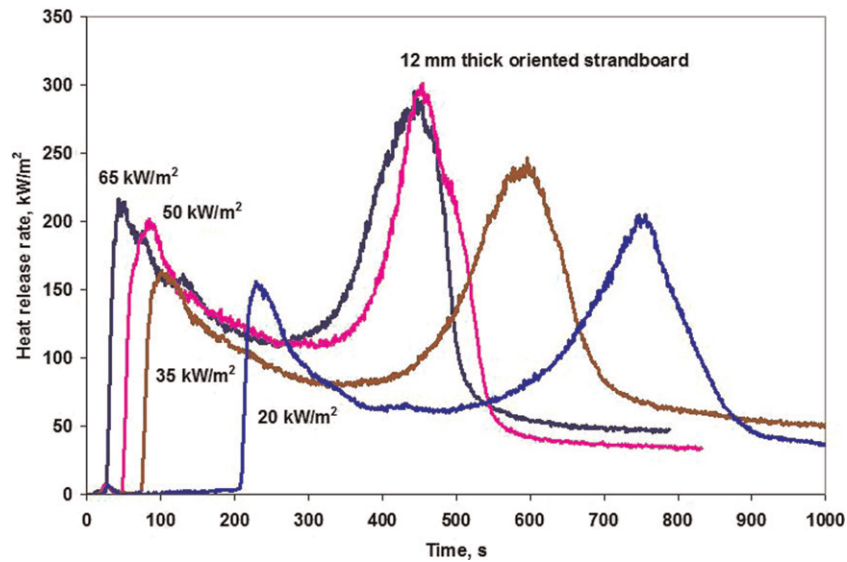


Figure 2 Heat release rate curves for 12 mm thick oriented strandboard (OSB) exposed to constant heat flux of 20, 35, 50, and 65 kW m⁻².

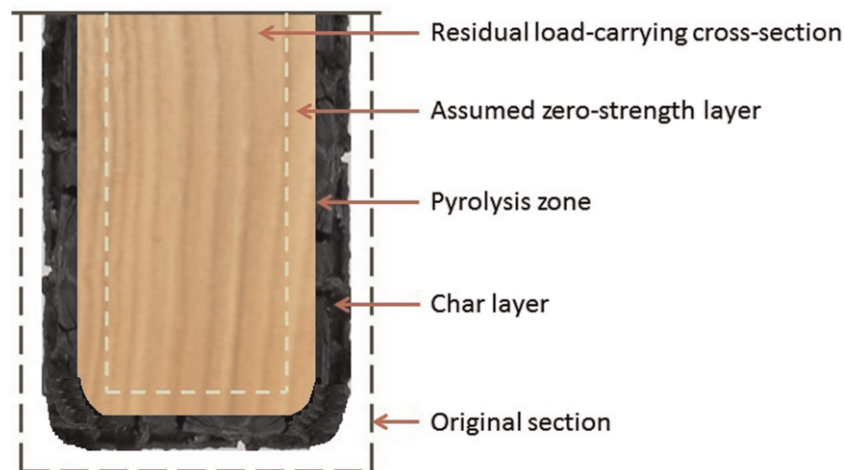


Figure 3 Illustration of char layer and zero-strength layer model of fire-damaged wood beam.

properties, which are affected by anatomical features. Charring in the longitudinal grain direction is reportedly double that in the transverse direction, and chemical composition affects the relative thickness of the char layer. Permeability affects the movement of moisture being driven from the wood or that being driven into the wood beneath the char layer.

The temperature at the innermost zone of the char layer is assumed to be 300 °C. Because of the low thermal conductivity of wood, the temperature 6 mm inward from the base of the char layer is about 180 °C. This steep temperature gradient means the remaining uncharred cross-sectional area of a large wood member remains at a low temperature and can continue to carry a load. Once a quasi-steady-state charring rate has been obtained, the temperature profile within the remaining wood cross-section can be used with other data to estimate the remaining load-carrying capacity of the uncharred wood during a fire and the residual capacity after a fire.

2.4 Flame Spread

Flame spread is the sliding movement of the flaming ignition point over the surface of a solid combustible. Thus, the flame foot provides the ignition source as well as mixing of volatiles and air. The quasisteady time response of the material to heat flux distribution from the flame and external sources in reaching the surface temperature of rapid volatilizations (coinciding with a point of ignition at around 300 °C depending on the wood species) determines the rate of flame spread. Rate of flame spread generally decreases with increases in density, moisture content, surface emissivity, surface temperature at ignition, and thermal

conductivity. It increases with the magnitude of heat exposure from the flame. This makes upward or ceiling flame spread (along the airflow direction) a serious fire safety issue because of the accelerating potential of room flashover with the attendant high rate of heat release and smoke and toxic gas production. In contrast, downward or lateral flame spread (opposite to airflow direction) has heating from the adjoining flame of such a small extent that external heat sources are required to sustain the flame spreading on wood products.

For most building applications, the regulatory test for building material acceptance in North America is the flame spread test known as the '25 ft' (7.6 m) tunnel (ASTM E84). Most untreated wood products have a Flame Spread Index of 60–190 in the ASTM E84 tunnel test. A wide range of flammability test methods is used for regulatory purposes worldwide and for research and development.

3 FRT of Wood

Most FRTs delay ignition, reduce heat release, and reduce flame spread (Rowell and Dietenberger, 2013). As discussed previously, many flame-retardant chemicals shift the thermal degradation to the low-temperature pathway of noncombustible gases and a greater proportion of char residue. The reactions are acid-catalyzed hydrolysis of the cellulose and hemicellulose. Chemicals dehydrate the wood and increase the condensation and cross-linking of the carbon skeleton. Other possible mechanisms for fire retardancy include formation of an insulating layer, increased dissipation via conduction of heat away from heat source, endothermic reactions to absorb heat, and release of radicals that inhibit flaming combustion.

For interior applications where leaching is not an issue, water-soluble inorganic salts are the common flame retardants. These salts include monoammonium and diammonium phosphate, ammonium sulfate, zinc chloride, sodium tetraborate, and boric acid. These chemicals are combined in formulations to develop optimum fire performance (flaming, smoldering, and smoke) yet still retain acceptable hygroscopicity, strength, corrosivity, and other properties. For example, boric acid is often added to reduce smoldering or glowing. Another chemical used in interior formulations is guanylurea phosphate. For a leach-resistant system, one method is to polymerize the fire-retardant resins after impregnation into the wood. Such a treatment is the amino resin system based on urea, melamine, dicyandiamide, and related compounds.

A major class of flame-retardant coatings includes those that intumesce to form an expanded low-density protective film upon exposure to fire. Intumescent formulations include a dehydrating agent, a char former, and a blowing agent. Potential dehydrating agents include polyammonium phosphate. Ingredients for the char former include starch, glucose, and dipentaerythritol. Potential blowing agents for the intumescent coatings include urea, melamine, and chlorate paraffin. Performance requirements can be found in the standards of the American Wood Preservers' Association. Flame-retardant-treated wood has a Flame Spread Index of less than 25 in ASTM E84 used in North America. As shown in Figure 4, FRTs generally reduce the initial peak heat release, average heat release, and the effective heat of combustion. Ignition times are often increased. Excessive levels of FRT can increase the production of smoke. FRTs tend to have minimal impact on charring rates and the fire endurance of structural members.

Wood treated with inorganic flame-retardant salts can be more hygroscopic than untreated wood. The increases in equilibrium moisture content will depend upon the chemical, level of chemical retention, and size and species of wood involved. A higher moisture content increases problems with corrosion of fasteners. Applications that involve high humidity require treated wood designed for low hygroscopicity. FRT of wood generally results in reductions in the mechanical properties of wood. Flame-

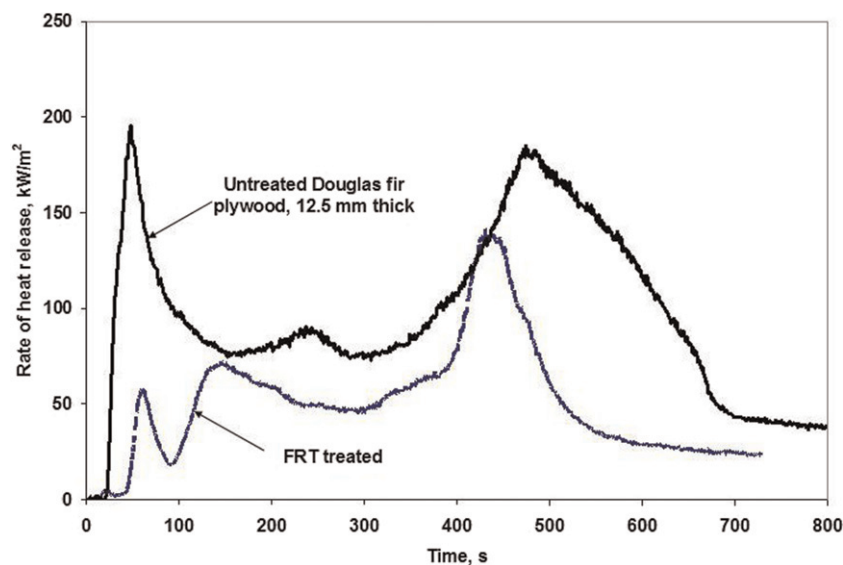


Figure 4 Heat release curves for untreated and fire-retardant-treated (FRT) Douglas-fir plywood, 12.5 mm thick.

retardant-treated wood is often more brittle than untreated wood. Some fire-retardant formulations can cause further losses in strength with continued exposure to elevated temperatures found in roof applications. Such losses occur when the fire-retardant salts catalyze the acid hydrolysis of the holocellulose at elevated temperatures much lower than that associated with a fire. The use of pH buffers in FR chemicals, such as borates, can partially mitigate the initial effect of the FR treatment on strength and reduce subsequent thermal degradation of mechanical properties.

4 Concluding Remarks

Issues of thermal degradation and fire performance are often overlooked in the use of wood because of its ease of use. In situations of elevated temperatures or direct fire exposure, an understanding of the thermal degradation and fire behavior of wood is needed. Although considerable research has been done on its thermal degradation and fire behavior, the complexity of the processes involved leaves many gaps in gaining a full understanding of thermal degradation and fire behavior of wood.

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