

FIRE RETARDANT PERFORMANCE OF SUGI AND HINOKI TREATED WITH PHOSPHORUS AND NITROGEN FIRE RETARDANT

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ABSTRACT

In this paper, ammonium phosphate polymer (APP), guanidinium phosphate urea (GUP), phosphonic acid, and a small number of additives that confer flame retardant properties were prepared as a new composite flame retardant. Cedar (*Cryptomeria japonica*) and hinoki (*Chamaecyparis obtuse*) penetrate and absorb the solution into the inner wall of the wood by vacuum pressurization, thus obtaining fire-retardant woods. The flame retardant effects at different absorption amounts were investigated by thermogravimetric analysis and cone calorimetry. The absorption amounts of both kinds of wood above 0.095 g cm^{-3} and 0.085 g cm^{-3} respectively, met the flame retardant standard ISO-5660-1: 2015. Thermogravimetric analysis showed that the fire-retardant-treated wood increased thermal stability, accelerated carbonization, and lower the decomposition temperature to below 300°C .

KEYWORDS: Combustion behaviors, fire retardant properties, cone calorimeter, thermal degradation, TG analysis.

INTRODUCTION

Wood is a natural material with low cost of production, low energy consumption, non-toxic, non-hazardous, and non-polluting, and is widely used as building materials and interior decoration materials. Due to its sustainable energy, high strength-to-weight ratio, thermal insulation, electromagnetic shielding, and acoustic proofing (Bi et al. 2021, Jancík et al. 2021, Samanta et al. 2022), as well as the promotion of energy conservation and emission reduction

and wood's decorative properties (good texture, environmental protection, high moisture absorption, and dehumidification ability, and good sensory), the demand for wood and wood products in residential and non-residential buildings is increasing. However, since wood and wood-based materials are primarily composed of cellulose, lignin, and hemicellulose, they are flammable and frequently cause avoidable fires that result in a huge number of casualties. As a result, enhancing the fire-retardant qualities of wood is an imperative but challenging step in wood applications (Wang et al. 2004, Di Blasi et al. 2007, Li et al. 2021, Samanta et al. 2022).

Fire retardants, as components of passive fire protection, play a critical role in wood applications. Many chemical alterations have been proposed to improve the fire resistance of wood while also improving its dimensional stability, mechanical qualities, and durability, including resin impregnation, silylation, in situ graft polymerization of monomers, and mineralization of wood tissue (Kawamoto et al. 2008, Zhou et al. 2011, Xie et al. 2014, Merk et al. 2016, Guo et al. 2017, Ratajczak et al. 2019, Pondelak et al. 2021). Traditional methods of halogen flame retardants and resin impregnation, on the other hand, produce toxic substances that are contrary to the concept of environmental friendliness. Some treatments, on the other hand, exploit synergistic interactions between boron, phosphorus, and nitrogen to enhance charcoal production, resulting in flame retardancy and significantly less hazardous chemicals. Traditional flame retardant systems, such as ammonium phosphate, boron, silica, or sulfur-based flame retardants, influence the pyrolysis process chemically by accelerating dehydration and carbonization, inhibiting the production of flammable volatile gases, or promoting the formation of an insulating coating or char layer. The charred coating inhibits the material's thermal breakdown and acts as a barrier to the passage of combustible decomposition products to the flame (Pan et al. 2014, Park et al. 2015, 2017, Kang et al. 2017, Zhu et al. 2020).

Fire protection can be obtained by depositing flame retardant additives as a coating or by impregnation, but the coating treatment method gradually renders the flame retardant effect ineffective over time, so impregnation of flame retardant chemicals into wood is one of the primary methods for improving wood's fire resistance (Zhang et al. 2020, Li et al. 2021). The most often utilized impregnants are water-soluble impregnants based on inorganic salts. They have low costs and low hazardous emissions, and they can also be used in ornamental and finishing layers (Wang et al. 2020).

Ammonium phosphate polymer (APP) and guanyl-urea phosphate (GUP) are proven effective fire-retardant chemicals that can lead to a lowering of the decomposition temperature and a higher char yield (Gaff et al. 2021). Guanidine and guanidine salts, in wood preservation, are mainly used as blowing agents in swelling paints and varnishes, while urea is in the majority of salts' fire retardants. Nitrogen compounds acting alone have limited fire-retardant effectiveness. Synergistic effects between P-N can increase the phosphorylation of cellulose (Xie et al. 2014, Salmeia et al. 2016, Zhang et al. 2016, Chu et al. 2017). Wang et al. (2004) synthesized a fire retardant for wood (FRW) with the main component of GUP and boric, and investigated the fire retardant synergism between them as well as the effect of the post-treatment drying method on the bonding strength of the wood treated with fire retardant chemical. Gao also synthesized a GUP and boric acid, and then determined the GUP compound fire retardant. And he further study the thermal degradation of wood treated with this guanidine compound (Gao 2003, Gao et

al. 2005). Harada et al. (2009) evaluated the fire-retardant wood treated with poly-phosphatic carbamate after the accelerated weathering test. Kong et al. (2018) combined treatment with biomass-derived furfuryl alcohol (FA) and ammonium dihydrogen phosphate (ADP) acting as a flame-retardant additive to prepare the wood/PFA/ADP composites. They found that the FA resin/ADP complexes increase phosphorus fixation in wood, which can provide long-term fire protection for wood in service. Wang et al. (2021) used ultrasonic impregnation to modify wood with guanidine dihydrogen phosphate (GUP) and zinc borate (ZB), resulting in strong thermal stability and flame retardancy of wood, and the results shown that wood/GDP/ZB had excellent smoke suppression qualities. Lin et al. (2021) treated wood synergistically with furfuryl alcohol (FA) and the flame retardant guanosine urea phosphate (GUP), and the treated wood not only has excellent dimensional stability and biological durability, but also has good heat resistance and fire resistance, indicating great potential for the development of outdoor finishing materials.

In this investigation, the Sugi (*Cryptomeria japonica*) and Hinoki (*Chamaecyparis obtuse*) were impregnated with new composite fire retardant chemicals consisting of APP, GUP, phosphoric acid as main components and a minor amount of additives. Using a cone calorimeter and the above-mentioned parameters, the fire-retardant performance with different chemical uptakes was analyzed, and the quantity required to meet the fire-retardant requirement for application was estimated. In addition, thermal degradation was studied using TG to learn more about the fire-retardant function following treatment with the new fire-retardant chemical.

MATERIALS AND METHODS

Materials

The chemicals used in this experiment are analytically pure. The chemicals included ammonium phosphate polymer, guanidyl urea phosphate, phosphonic acid, acrylamide-acrylic acid-N-methacrylamide copolymer, 2-benzisotiazolin-3-one, sodium hydroxide and hydrochloric acid. Wood were obtained from Japanese sugi (*Cryptomeria japonica*) and Hinoki (*Chamaecyparis obtuse*). Plain wood boards with no meson and no defects of close weight were selected and dried to 8% moisture content (MC) before treatment, labeled, and weighed.

Treatment of wood specimens

Sugi (*Cryptomeria japonica*) and Hinoki (*Chamaecyparis obtuse*) were cut into 910 mm (longitudinal) × 100 mm (tangential) × 10 mm (radial) samples, to which flame retardant treatments were applied. The main components of water soluble fire retardant solution are APP, GUP, phosphonic acid, acrylamide-acrylic acid-N-methacrylamide copolymer, 2-benzisotiazolin-3-one and a minor amount of additives, with a 25% concentration of the fire retardant, a specific gravity of 1.13 (20 ± 2°C) and a pH of 7.6 (20 ± 2°C).

Wood samples were first placed randomly in a vertical cylindrical container. To allow air to be removed from the wood, a vacuum of -0.098 MPa was applied to the container for 10 min. Afterward, the flame retardant agent was returned to the vessel, the pressure was increased to 1.5 MPa and continued for 30 min, and the pressurization process was repeated. The successful impregnation of the pharmaceuticals into the wood samples was determined based on

the difference in mass between the pretreated boards and before and after treatment of the boards. Finally, the impregnated treated wood samples were air-dried for two weeks and the moisture content was dried to 12% at 60°C addition, which means that the wood samples were successfully treated with flame retardant (Fig. 1).

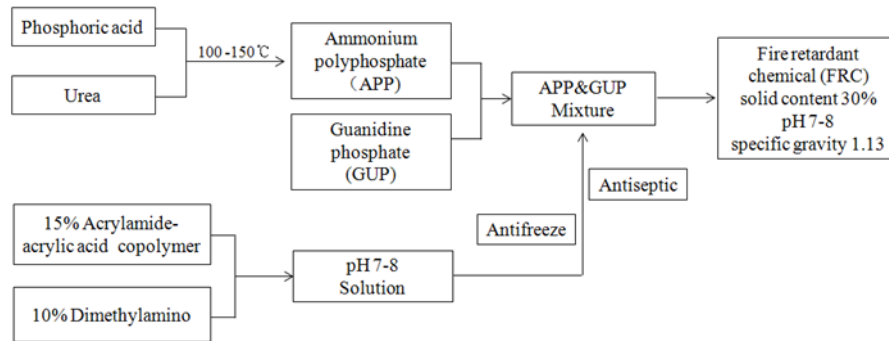


Fig. 1: Synthesis process of water-soluble phosphorus fire retardant (WPF).

Chemical uptake

The amount of substance of chemically absorbed flame retardant (FR) of the wood sample is calculated as shown below:

$$\text{Absorption (g cm}^{-3}\text{)} = (M_t - M_u)/V \quad (1)$$

where: M_t (g) - the mass of the sample after flame retardant treatment, M_u (g) - the mass of the sample without flame retardant treatment, V (cm³) - the volume of the wood sample, and $M_t - M_u$ (g) is also the mass of the substance of chemically absorbed FR.

Void volume filled (VVF)

Based on the assumption that within the given volume of the sample with known basic density and constant cell wall density (1500 kg m⁻³), there will be a void volume composed of cell lumina, intercellular spaces, and penetrable cell wall voids, which can be filled by liquids (Park et al. 2015, Salmeia et al. 2016). The porosity as void volume (P) and void volume filled (VVF) of wood samples were calculated as follows:

$$P = \left(1 - \frac{SG_t}{1.50}\right) \times 100 \quad (2)$$

$$VVF = \left(\frac{M_t - M_u}{V \times P}\right) \times 100 \quad (3)$$

where: SG_t is the specific gravity of the treated sample, M_t (g) is the mass of the treated sample, and M_u (g) is the mass of the untreated sample. V (cm³) is the sample volume before oven-dried to 12% MC.

Thermogravimetry analysis (TGA)

Thermal degradation properties of the samples were examined using a thermogravimetric analyzer SDT-Q600 V20.9 made by TA Instruments, USA. The measurements were taken in a dynamic nitrogen (dried) atmosphere with a temperature increase rate of $10^{\circ}\text{Cmin}^{-1}$, and the changes in the samples were recorded at an ambient temperature of 600°C . The sample was pulverized to a mesh size of 50 and weighed approximately 5 mg.

Cone calorimetric test

The increase of the sample was set to about 150 g, 250 g, 350 g, and 550 g samples, and the absorption was set from 0.04 g cm^{-3} to 0.163 g cm^{-3} . The cone calorimetry method was used to refer to the ISO-5660-1: 2015 standard, and the sample plane size was set to $100 \times 100\text{ mm}$, the radiation power was 50 kW m^{-2} , and the heating time was 10 min, and the samples were divided into four groups of repetitive tests to determine the total heat release (THR) and smoke generation rate (SPR) of the samples, so as to analyze the flame retardant and smoke suppression performance of the materials and accurately evaluate the combustion performance of the materials.

RESULTS AND DISCUSSION

TGA

Based on the above analysis, Sugi and Hinoki wood samples with the uptake of 0.095 g cm^{-3} and 0.085 g cm^{-3} were fixed for TGA to investigate the thermal stability of both wood species with WPFRT treatment. The thermal degradation data for Sugi and Hinoki are listed in Tab. 1.

Tab. 1: Thermal degradation data of Sugi and Hinoki samples by TGA.

Sample	Stage	Temperature ($^{\circ}\text{C}$)	Mass loss (%)	DTG max ($^{\circ}\text{C}$)	Residue (%)
S-C	Drying step	216-326	29	346	17.20
	Charring step	326-408	38		
	Calcining step	408---	12		
S-T	Drying step	149-244	14	272	30.60
	Charring step	244-417	33		
	Calcining step	417---	20		
H-C	Drying step	221-309	29	365	14.39
	Charring step	309-422	43		
	Calcining step	422----	9		
H-T	Drying step	176-232	10	265	33.50
	Charring step	232-388	30		
	Calcining step	388---	22		

Figs. 2 and 3 show the TGA and derivative thermogravimetric (DTG) curves of the samples. The TGA curves demonstrate that the untreated and treated wood samples lost weight in three stages while being heated: the drying stage, the charring stage, and the calcination stage. The important information for the treated and untreated wood samples at the three stages is summarized in Tab. 1. According to the findings, the rapid decomposition temperatures of Sugi

and Hinoki decreased from 346°C to 272°C and from 365°C to 265°C, respectively. Meanwhile, the mass residue increased to more than 30%. The drying phase of untreated Sugi and Hinoki occurred at about 216-326°C and 221-309°C respectively, as a result of the release of moisture absorbed by both kinds of wood during storage and experiments, and during the charring phase mainly as a result of the decomposition of cellulose and hemicellulose (Guo et al. 2018), leading to a mass loss of 38% and 43% respectively, which is the main degradation phase of thermal decomposition. As the warming process continued, the weight gradually dropped at around 408°C and 422°C. When the remaining components were mostly charcoal, the weight gradually decreased as the heating process continued, and the lignin broke down into cross-linked aromatic charcoal with mass residues of 17.2% and 14.39%, respectively (Zhang et al. 2021).

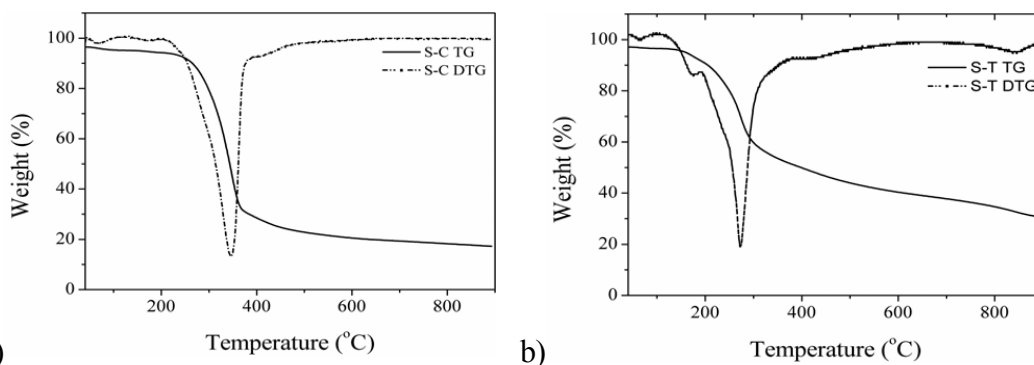


Fig. 2: TG and DTG curves of Sugi wood without (a) and with treatment (b) (S-C: Sugi control; S-T: Sugi treatment).

The data suggest that the addition of chemicals to the WPFW-treated Sugi and Hinoki during the drying stage may encourage heat absorption and hasten water evaporation, which could lead to pyrolysis of both types of wood early. In the main degradation stage (charring step), due to the release of phosphoric acid from the samples, these released acids catalyze the decomposition or dehydration of hemicellulose, and APP decomposition produces phosphoric acid, HPO and PO radicals, which are considered as free radical scavengers. Thus, HPO and PO can trap the free radicals produced by the wood. This leads to the early onset of thermal degradation of the wood. The results show that the addition of flame retardant chemicals leads to earlier onset of thermal degradation of the wood and promotes char formation. The residual amount of both treated woods was also higher than that of untreated wood, when the endpoint temperature was reached, which was attributed to the char formed during the charring process of the wood absorbing some noncombustible gases and adsorbing some incomplete combustion residue chemicals. The addition of GUP is also effective in promoting the formation of a charcoal layer, which creates a barrier layer to protect the wood (Lin et al. 2021). Sugi and Hinoki each had a residual quantity of 30.6% and 33.5%, respectively.

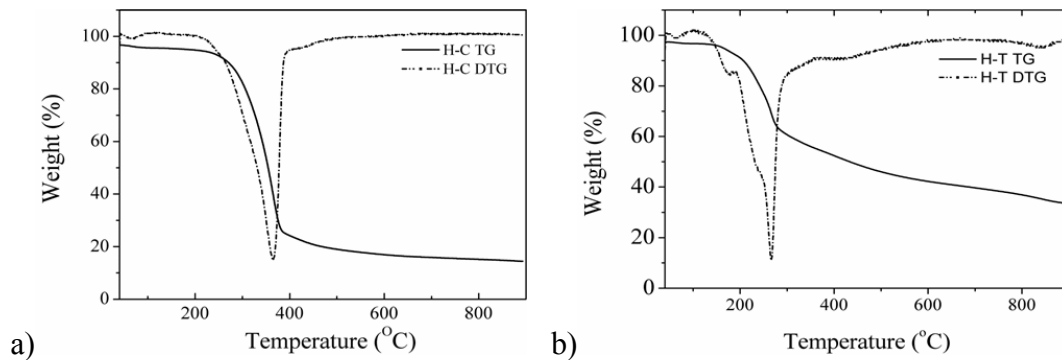


Fig. 3: TG and DTG curves of Hinoki wood without (a) and with treatment (b) (H-C: Hinoki control; H-T: Hinoki treatment).

The samples of sugi and hinoki treated with APP, GUP, and phosphonic acid showed the following principal alterations as a result of thermal deterioration: (1) The addition of WPFR, which phosphorylated and catalyzed the dehydration of the samples and accelerated the thermal disintegration of the wood, may have caused the initial temperature of thermal degradation to be advanced. (2) The principal thermal degradation temperatures of sugi and hinoki, as shown by the DTG curves, were advanced by 74 and 100°C, resp. This finding suggests that the flame retardant flame retardant chemicals catalyzed the decomposition of wood, causing thermal decomposition to occur below 300°C. (3) The increase in the final mass residue of thermal degradation indicates the formation of residual carbon, which can effectively impede the heat and flame transfer, thus achieving the flame retardant effect.

Cone calorimetry test

Heat release

The THR and HRR topical curves of samples were shown in Fig. 4. The THR at 300 s, peak of HRR, and the time of peak of heat release rate (pkHRR) are given in Tab. 2. The THR and HRR measurements are the quantity of most concern in predicting the course of the fire and its effect. The slope of the THR curve can be assumed as representative of fire spread. The lower the THR value, the safer the material. As shown in Fig. 4, the slope of samples decreased as fire-retardant (FR) uptake increased. When the uptake was up to 0.095 g cm⁻³ and 0.085 g cm⁻³ for Sugi and Hinoki, THR values were below 8 MJm⁻² at 300 s.

Tab. 2: Combustion parameters of Sugi and Hinoki samples with different WPFR uptake.

Sample	Uptake (g cm ⁻³)	VVF (%)	THR (300 s) (MJm ⁻²)	pkHRR (kW·M ⁻²)	Time of pkHRR (s)	Fire growth index (FGI)
Sugi-1	0.058	13.02	19.57	139.39	336	0.41
Sugi-2	0.073	16.38	11.81	97.93	406	0.24
Sugi-3	0.095	22.08	5.88	46.21	378	0.12
Sugi-4	0.135	33.98	8.26	76.25	282	0.27
Hinoki-1	0.040	9.98	21.00	161.23	358	0.45
Hinoki-2	0.065	17.43	20.72	179.93	362	0.50
Hinoki-3	0.085	25.89	3.99	51.07	358	0.14
Hinoki-4	0.163	47.69	2.32	94.26	434	0.22

Note: VVF, void volume filled; THR, total heat release; pkHRR, the peak of heat release rate.

The WPFR FR chemicals containing phosphorus can release phosphoric acid which catalyzes the dehydration and carbonization of wood, resulting in the formation of less flammable products and correspondingly more char. In this fire-retardant system, APP and GUP, as P-N containing chemicals, acted as the acid source and blowing agent. The fire retardant formed a carbonaceous layer on the surface of wood by dehydration, and the formation of double bonds, thus initiating cyclization and cross-linking.

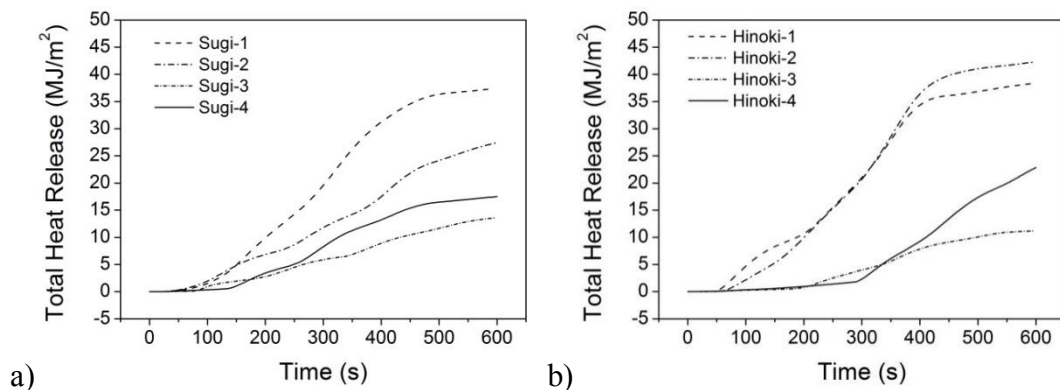


Fig. 4: Total heat release (THR) curves of Sugi (a) and Hinoki (b) samples with different WPFR uptakes.

In Tab. 2, the fire growth index (FGI) is the ratio of peak heat release rate (pkHRR) to peak time. The higher the FGI is, the faster for material to catch fire and flame to spread. Opposite, lower FGI means safer material in the fire condition. At the basic uptakes that meet fire requirements 0.095 g cm^{-3} and 0.085 g cm^{-3} for Sugi and Hinoki, it was found that the values of pkHRR were lower, and the time of pkHRR was prolonged, which resulted in a smaller FGI.

Gas and smoke release

Smoke production rate (SPR) is the smoke produced instantaneously and calculated by multiplying a specific extinction area by the mass-loss rate. It is also one of the most important parameters representing fire risk related materials (Cong et al. 2018). The SPR of samples with different FR uptake as a function of time is shown in Fig. 5. Smoke is formed first at the beginning of burning and decreases before the end of burning. The first main peak was formed at the beginning of burning, and the second main peak appeared before the end of burning. In addition, the majority of smoke was produced during flame combustion. The fire-retardant treatment can decrease the pyrolysis, thus the pyrolysis product can be burned completely. And consequently, the smoke decreased. The effect of the fire-retardant chemical on smoke is apparent. The first peak was decreased by 63% and 72%, compared with the uptake below 0.095 g cm^{-3} and 0.085 g cm^{-3} for Sugi and Hinoki. This may be attributed to the competition between the char-forming fire retardant action and the evolution of combustible gases. When pine wood was heated, GUP and APP decomposed and then produced incombustible materials such as NH_3 and CO_2 to a barrier or dilute oxygen of combustible gases.

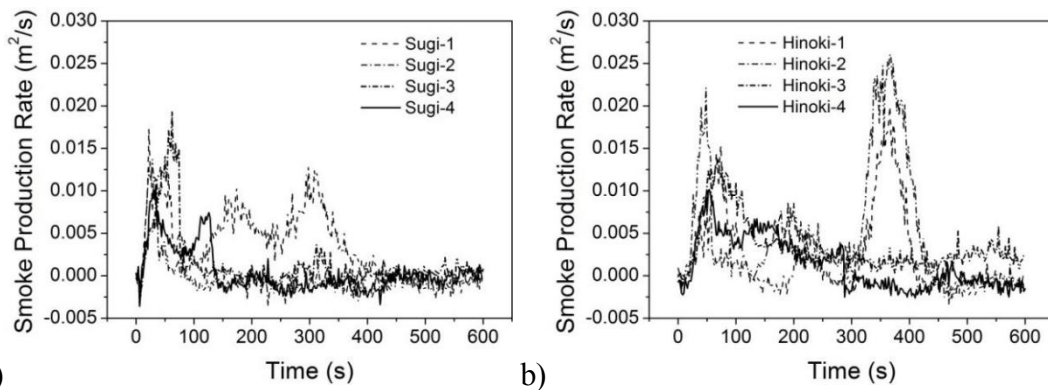


Fig. 5: Smoke production rate (SPR) curves of Sugi (a), Hinoki (b) samples with different fire-retardant uptakes.

CONCLUSIONS

(1) According to the results of the cone calorimetry test, the flame retardant properties of Sugi and Hinoki were significantly improved after the flame retardant treatment, as evidenced by the fact that both treated woods were able to meet the flame retardant requirements (THR less than 8 MJ m^{-2}) when the WPFR absorption of Sugi and Hinoki was 0.095 g cm^{-3} and 0.085 g cm^{-3} and above, respectively. (2) According to the results of thermogravimetric analysis, the wood treated with APP, GUP, and phosphonic acid as the main components of flame retardants can effectively promote the dehydration of wood into charcoal during combustion, as well as improve the thermal stability of wood and lower the decomposition temperature to below 300°C . The results demonstrate that adding intumescent flame retardants can generate a char layer on the surface of the substrate, inhibiting the transfer of oxygen heat and reducing the creation of flammable materials and that the flame retardant performance has been considerably enhanced.

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