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THE EFFECT OF COMBINED FLAME RETARDANTS ON THE PHYSICAL AND MECHANICAL PROPERTIES OF MONGOLIAN PINE

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ABSTRACT

To enhance the flame-retardant properties and physical-mechanical performance of wood, this experiment selected Mongolian pine as the material and employed a vacuum-pressure impregnation method, with different ratios of borax and ammonium polyphosphate (BO:APP) (1:1, 2:1, 1:2) applied and compared with untreated wood (BCW). The treated and untreated Mongolian pine samples were analyzed using scanning electron microscopy (SEM), a universal mechanical testing machine, a limiting oxygen index (LOI) tester, and thermogravimetric analysis (TG) to assess microstructure, weight gain rates, density, physical-mechanical properties, flame retardancy, and thermal stability. Results indicate that the flame retardants were uniformly dispersed within the wood's pores, achieving excellent impregnation. The weight gain rates and density of the treated wood improved when the weight ratio of borax to ammonium polyphosphate was in ratio1:1 (BO-1:APP-1 group).The weight gain rates was 10.36% and a density of 0.632 cm^3/g and the MOE and MOR reached 12,076 MPa and 116.3 MPa, respectively, representing increases of 26.5% and 16.7% compared to untreated samples. The oxygen index of the BCW group was 23.1%, while that of the BO-1:APP-1 group was 42.5%, reflecting an 84% improvement over the BCW group. The thermal decomposition temperature of the treated samples decreased by 50°C, with the BCW group's char yield at 11.35%, whereas the char yield for treated samples exceeded 20%, reaching 37.44% for the BO-1:APP-1 group, marking a 230% increase compared to the BCW group.

KEYWORDS: Composite flame retardants, Mongolian pine, mechanical properties, flame retardant properties, thermogravimetric analysis.

INTRODUCTION

Wood, as a time-honored building material and raw material for furniture production, still occupies an indispensable position in modern society. Its flammability, however, limits its application in fields with high fire safety requirements (Zhu et al. 2014, Chen et al. 2024, Zhang et al. 2024). The use of flame retardants has become an effective means to enhance the flame retardancy of wood. Traditional single flame retardants can improve the flame retardancy of wood to some extent, but they often suffer from limited effectiveness and poor environmental friendliness. Therefore, the research and development of combined flame retardants, with their high efficiency and environmentally friendly characteristics, have become a focus in the field of wood flame retardancy (Xie et al. 2024, Jia 2021).

Combined flame retardants have shown significant advantages in enhancing the flame retardancy of wood. Currently used flame retardants mainly include phosphates (Fan et al. 2024), phosphorus-nitrogen compounds (Wu et al. 2023), borates (Xiao et al. 2022), and organic flame retardants (Gao et al. 2024). However, their impact on the mechanical properties of wood remains controversial. Flame retardant treatment may reduce the physical strength and toughness of wood, which is an unfavorable factor for the ultimate application of wood (Hao et al. 2016, Wang et al. 2017, Pan 2024). Research on how to maintain the flame retardancy of wood while minimizing the impact on its mechanical properties is a key research topic.

Ammonium polyphosphate (APP) is a new type of efficient, halogen-free flame retardant widely used in the flame retardant field (Fan et al. 2016). Shi (2017) combined APP with boric acid/borax mixtures and ammonium chloride to prepare samples by adjusting the components and proportions, and studied the flame retardancy of the treated pine wood samples prepared by the atmospheric pressure impregnation method. Tian et al. (2023) and others applied trifluorophenyl isocyanate (TBC) to the core layer and ammonium polyphosphate (APP) to the surface layer. By adjusting the addition ratio of APP to TBC, the mechanical properties and flame retardancy of the treated plywood were controlled. Hu et al. (2023) synthesized a decorate ammonium polyphosphate-based flame retardant (MAPP-CA) with ammonium polyphosphate (APP), ethylenediamine, and caffeic acid.

Sodium tetraborate decahydrate (borax, BO), as one of the important boron compounds, can be used alone or as an auxiliary agent in combination with phosphorus-based, nitrogen-based, and other flame retardants, widely used in polymers, cedar, and wood products (Donmez et al. 2015, Dogan et al. 2021). Cui et al. (2018) conducted experimental research on the combustion performance and pyrolysis characteristics of Yunnan pine treated with ammonium dihydrogen phosphate and borax synergistic flame retardants. Tan et al. (2022) crushed natural (organic) oyster shells (*Chamelea gallina*) and made a solution with water, which was used alone or in combination with boron compounds (borax) after the pyrolysis process to measure the oxygen index of the impregnated wood. Kumar et al. (2022) initially treated bamboo fibers with alkali (NaOH), boric acid borax (Ba-Bx), and borax (Bx). The treated and untreated fibers were compounded with ammonium polyphosphate (APP) to study their synergistic effects on thermal stability, flame retardancy, and mechanical properties.

This study aims to deeply explore the effects of combined flame retardants of ammonium polyphosphate and borax on the flame retardancy and physical-mechanical properties of *Pinus sylvestris* var. mongolica, in order to find an effective method. Through experimental analysis, this study will evaluate the specific impacts of different combined flame retardant treatments on the flame retardancy and mechanical properties of *Pinus sylvestris* var. mongolica wood, and explore the underlying mechanisms. In addition, this study will also compare the effects of different flame retardant formulations to determine the optimal flame retardant combination, providing scientific basis and technical guidance for practical applications.

MATERIALS AND METHODS

Materials

Mongolian pine (*Pinus sylvestris var. mongolica*), 400 mm (long) × 50 mm (tang) × 25 mm (rad) samples, produced in Northeast China. Ammonium polyphosphate (APP) provided by Shanghai Jieshi Kai Biotechnology Co., Ltd. Borax (BO) provided by Fuchen (Tianjin) Chemical Reagents Co., Ltd. All reagent used analytical grade. Deionized water, homemade by the College of Material Science and Art Design, Inner Mongolia Agricultural University.

Preparation of compound flame retardant

This study established multiple experimental groups to investigate the effects of different flame retardant formulations on the flame retardancy of *Pinus sylvestris*. The specific configurations are as follows: *(1)* Control group: No flame retardant was added, serving as a blank control (BCW). *(2)* Single flame retardant groups: Borax group and ammonium polyphosphate group: 200 g of borax (BOW) and 200 g of ammonium polyphosphate (APPW) were weighed separately using an electronic balance and dissolved in 1800 mL of distilled water, respectively, to obtain a 10% borax solution and a 10% ammonium polyphosphate solution. *(3)* Compound flame retardant groups (borax: ammonium polyphosphate): The borax and ammonium polyphosphate were mixed in three different mass ratios: 1:1 (BO-1:APP-1), 2:1 (BO-2:APP-1), and 1:2 (BO-1:APP-2). In each case, the total amount of borax and ammonium polyphosphate was 200 g, which was dissolved in 1800 mL of distilled water to obtain a 10% composite flame retardant solution. The key emphasis is on the mass ratios of borax to ammonium polyphosphate: 1:1, 2:1, and 1:2.

Preparation of flame-retardant pine wood

To ensure consistency in the experiment, the *Pinus sylvestris* specimens were first cut into uniform sizes using a fine woodworking band saw: 36 cubes of 20 mm (L) × 20 mm (T) × 20 mm (R), 42 wooden bars of 300 mm (L) × 20 mm (T) × 20 mm (R), 36 wooden strips of 120 mm (L) × 10 mm (T) × 4 mm (R), and 6 wooden strips of 20 mm (L) × 10 mm (T) × 10 mm (R). An electronic balance was used with the weight error controlled within 0.1 g.

The *Pinus sylvestris* samples were treated using the vacuum-pressure impregnation method. After immersion in the flame retardant solution, the samples were placed in a vacuum-pressure tank with a vacuum level of 0.1 MPa, a vacuum duration of 20 min, a pressure of 0.9 MPa, and a pressure duration of 30 min to ensure thorough penetration of the flame retardant. The liquid level was maintained 1 cm above the samples to ensure uniform impregnation. After impregnation, the samples were dried in an oven at 70°C until a constant weight was achieved (Shi 2017). Following drying, one 300 mm (L) × 20 mm (T) × 20 mm (R) sample from each group was cut into small strips, pulverized using a grinder, and passed through a 100-mesh sieve. Approximately 1 g of wood flour was collected for thermogravimetric analysis. This procedure was repeated three times for each group of samples (Guan et al. 2016).

Scanning electron microscopy (SEM)

The morphology of flame-retardant wood was observed using the Phenom-S4800 scanning electron microscope from Carl Zeiss Microscopy (Czech Republic). Prior to testing, the sample was attached to conductive adhesive and coated with a thin layer of metal. The coating thickness was 3-30 nm, with an operating voltage of 10 kV and a magnification of 400X.

Weight gain rates

Standardized specimens with dimensions of 20 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) were used to test the weight gain rates of the impregnated wood blocks. The weight gain rates of the impregnated and modified material can be calculated using as:

$$WPG = \frac{m_a - m_0}{m_0} \times 100\% \tag{1}$$

where: m_0 - dry weight of the sample before impregnation (g), m_a - dry weight of the sample after impregnation (g).

Density

This study followed the test method outlined in GB/T 1927.5-2021 "Test methods for physical and mechanical properties of defect-free small samples of wood. Part 5: Determination of density" to measure the density of the wood. The oven-dry density can be calculated as:

$$\rho_0 = \frac{m_0}{V_0} \tag{2}$$

where: ρ_0 - oven-dry density of the sample (g/cm^3), m_0 - oven-dry mass of the sample (g), v_0 - oven-dry volume of the sample (cm^3).

Modulus of rupture (MOR) and bending modulus of elasticity (MOE)

Using standardized specimens measuring 300 mm (L) × 20 mm (T) × 20 mm (radial), the modulus of rupture (MOR) (Eq. 3) and bending modulus of elasticity (MOE) (Eq. 4) of the samples were measured using the WDW-200 microcomputer-controlled electronic universal testing machine from Jinan Tianchen Testing Machine Manufacturing Co., Ltd. The test followed the procedures outlined in GB/T 1927.9-2021 "Test methods for physical and mechanical properties of defect-free small samples of wood. Part 9: Determination of modulus of rupture" and GB/T 1927.10-2021 "Test methods for physical and mechanical properties of defect-free small samples of wood. Part 10: Determination of bending modulus of elasticity":

$$\sigma_{b,W} = \frac{3P_{max}l}{2bh^2} \tag{3}$$

$$E_W = \frac{23Pl^3}{108bh^3f} \tag{4}$$

where: $\sigma_{b,w}$ - modulus of rupture, (MPa), P_{max} - maximum failure load, (N), E_W - bending modulus of elasticity, (MPa), P - difference between the upper and lower load limits, (N), l - span between the two supports, (mm), b - width, (mm), h - height, (mm), f - deformation between the upper and lower load limits, (mm).

Oxygen index analysis

A specimen with dimensions of 120 mm (L) × 10 mm (T) × 4 mm (R) was used, and the limiting oxygen index (LOI) was measured with the JF-3 Oxygen Index Apparatus produced by Jiangning Analytical Instrument Factory in Nanjing, following the standard GB/T 2406.3-2022 "Plastics. Determination of combustion behaviour by oxygen index method. Part 3: High temperature test".

Thermogravimetric analysis (TGA)

A sample of 20 mg of uniformly textured powder was subjected to thermogravimetric analysis using a Netzsch STA 449 F5 simultaneous thermal analyzer from Shenzhen Keshida Electronic Technology Co., Ltd. The temperature range for the test was set from 30°C to 800°C, with a heating rate of 10°C/min, under a nitrogen atmosphere, and a gas flow rate of 20 mL/min.

RESULTS AND DISCUSSION

SEM analysis

Fig. 1 SEM images of various experimental groups. Fig. 1a displays the micro-structure of the blank control group, which has a loose internal structure with numerous pores. Fig. 1b and 1c show the micro-structure of the samples treated with boric acid and ammonium polyphosphate, respectively.

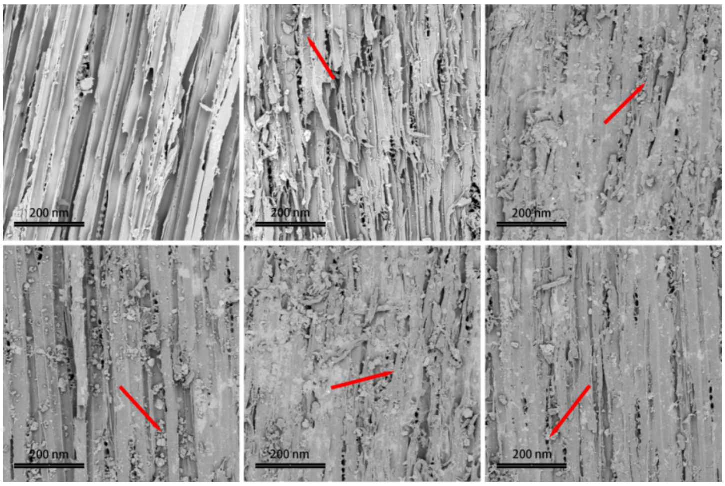


Fig. 1: (a) BCW, (b) BOW, (c) APPW, (d) BO-1: APP-1, (e) BO-2: APP-1, (f) BO-1: APP-2 micro-structural images of samples.

As indicated by the red arrows in the figures, it can be seen that boric acid and ammonium polyphosphate are filled within the wood cells. Figs. 1d,e, and f present the micro-structure of the samples treated with boric acid and ammonium polyphosphate in different proportions. Compared to the samples treated with single components, the samples treated with the combined flame retardants exhibit a richer filling material, which is abundantly deposited within the wood cells.

Weight gain rates and density

Fig. 2 is a graph showing the analysis of weight gain rates and densities for samples treated with different flame retardants. In Fig. 2, BCW represents the untreated specimens. The graph illustrates that the weight gain rates of the specimens after impregnation treatment generally ranges around 10%, with the BO-1:APP-1 treatment exhibiting the best performance, achieving a weight gain rates of 10.36%. Additionally, the combined impregnation treatments show superior results compared to single-component impregnation, a conclusion that is also supported by the SEM images above. The density of the BCW specimens is 0.582 g/cm³, whereas the density of the impregnated specimens is greater than 0.6 g/cm³, indicating a significant increase in density compared to the untreated specimens.

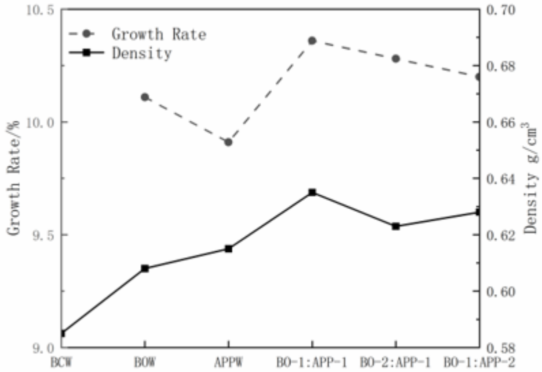


Fig. 2: Comparison of weight gain rates and density for different flame retardant treatments.

Modulus of rupture (MOR) and bending modulus of elasticity (MOE)

Fig. 3 is an analysis chart of MOR and MOE for specimens treated with different flame retardants. The MOE and MOR values of wood can provide a general indication of its physical and mechanical properties (Li et al. 2024).

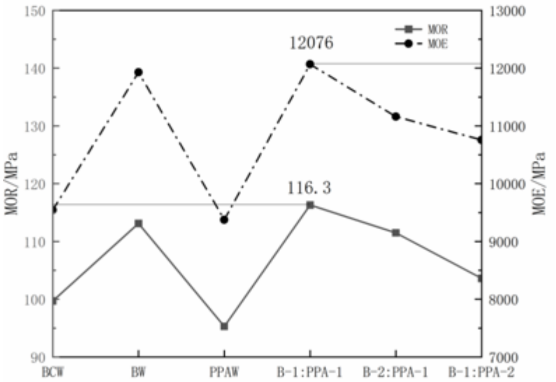


Fig. 3: MOR and MOE of specimens treated with different flame retardants.

As observed in Fig. 3, the MOR and MOE of the specimens treated with flame retardants, except for those treated with APP, have shown improvement. The specimens treated with the BO-1:APP-1 mixture reached MOE and MOR values of 12076 MPa and 116.3 MPa, respectively, which represents an enhancement of 26.5% and 16.7% over the untreated specimens. It can be concluded that the mechanical strength of the flame-retardant treated specimens, excluding those treated with APP, has increased, with the BO-1:APP-1 mixture demonstrating the most superior mechanical performance.

Oxygen index

Fig. 4 is a graph of the oxygen index for specimens treated with different flame retardants. The limiting oxygen index (LOI) refers to the minimum oxygen concentration in a mixture of nitrogen and oxygen at which a material can sustain flame combustion. An oxygen index below 22% indicates that the sample is highly flammable, while an index between 22% and 27% classifies the material as combustible. An oxygen index above 27% signifies flame-retardant wood; the higher the oxygen index, the less susceptible the wood sample is to combustion, indicating better flame-retardant properties (Jiang et al. 2016). In Fig. 4, it can be observed that the BCW group commenced burning at an oxygen index of 23.1%. The oxygen indices of the flame-retardant treated specimens were all above 30%, and after combined treatment, the oxygen indices of the specimens were all greater than 35%. The BO-1:APP-1 group exhibited the highest oxygen index at 42.5%, which represents an 84% increase compared to the BCW.

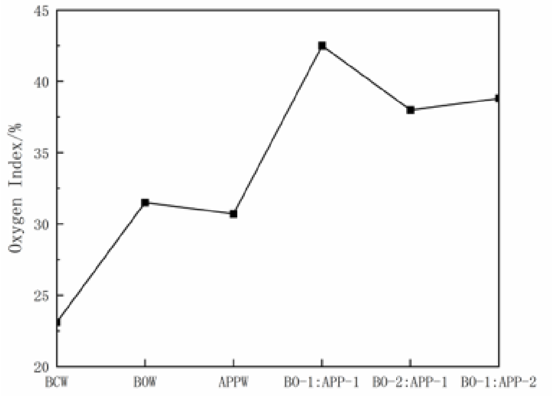


Fig. 4: Oxygen index of specimens treated with different flame retardants.

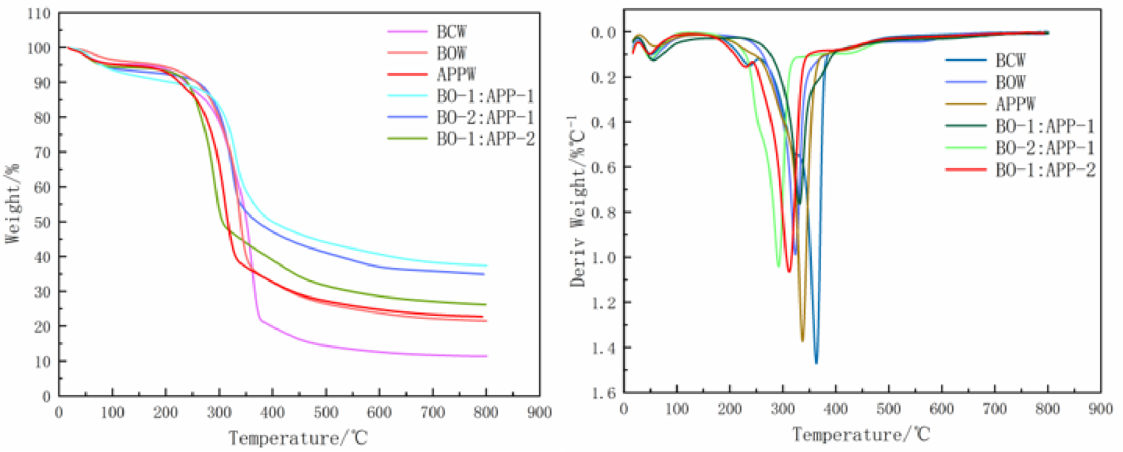
TGA

Tab. 1 shows the thermal degradation data of Mongolian pine before and after flame retardant treatment.

Tab. 1: Thermal degradation data before and after flame retardant treatment obtained by TGA.

Sample	Stage	Temperature (□)	Mass loss (%)	DTG max (□)	Residue (%)
BCW	Drying step	187-322	26	369	11.35
	Charring step	322-450	52		
	Calcining step	450---	6		
BOW	Drying step	147-278	14	311	24.22
	Charring step	278-411	33		
	Calcining step	411---	21		
APPW	Drying step	201-335	16	318	21.76
	Charring step	335-414	34		
	Calcining step	414---	18		
BO-1:APP-1	Drying step	252-338	9	279	37.44
	Charring step	338-469	30		
	Calcining step	469---	19		
BO-2:APP-1	Drying step	203-298	11	286	35.13
	Charring step	298-487	32		
	Calcining step	487---	19		
BO-1:APP-2	Drying step	206-315	13	304	27.35
	Charring step	315-420	33		
	Calcining step	420---	22		

Figs. 5 show the TGA and derivative thermogravimetry (DTG) curves of the samples. In thermogravimetric analysis (TGA) tests, the thermal decomposition process of wood is typically divided into three stages: drying stage, carbonization stage, and calcination stage. From 0 to 300°C, the evaporation of free water and bound water in the wood leads to a decrease in mass, which is the drying stage. From 300 to 500°C, the organic substances in the wood begin to decompose, forming charcoal and other volatile products, which is the carbonization stage. From 500 to 800°C, the remaining carbonized materials are oxidized, resulting in the formation of gases and ash, which is the calcination stage (Wang et al. 2021). The information of the three stages for both treated and untreated wood is presented in Tab. 1.



a) *b)*

Fig. 5: a) TG (Thermogravimetric) curves, b) DTG (Derivative Thermogravimetric) curves of specimens treated with flame retardants.

Based on the research findings, the rapid decomposition temperature of the untreated wood was found to be 369°C. The rapid decomposition temperature of the treated wood was reduced by more than 50°C compared to the untreated wood, and the rapid decomposition temperature of the BO-1:APP-1 group was decreased by 90°C compared to the untreated wood. Additionally, the mass residue increased by more than 20%. The drying stage of the untreated Mongolian pine occurred at 187-322°C, during which the mass reduction was primarily due to the evaporation of free water and bound water in the wood. The carbonization stage mainly resulted from the decomposition of cellulose and hemicellulose (Guo et al. 2018), which is the primary degradation stage of thermal decomposition. As the heating process continued, the weight gradually decreased. When the remaining components were predominantly charcoal, the weight gradually reduced with ongoing heating, as lignin decomposed into cross-linked aromatic carbon (Zhang et al. 2021). The BCW group began to pyrolyze at 300°C, whereas the specimens treated with flame retardants started to pyrolyze around 250°C, indicating that the pyrolysis stage of the flame-retardant-treated specimens was advanced by 50°C compared to the BCW group. During this period, dephosphorization and thermal decomposition occurred, and the addition of flame retardants accelerated the pyrolysis process of the wood (Pintor-Ibarra et al. 2024). The peak height of the specimens represents the stability of the sample, and the peak area indicates the magnitude of mass change. From Fig. 6, it can be observed that the stability of the flame-retardant-treated specimens increased, and the weight loss variation decreased. This is due to the dephosphorization reaction of APP during the pyrolysis process, which releases volatile inert gases that dilute the combustible gases, while the non-oxide compounds generated by boric acid cover the surface, acting to some extent as a barrier to oxygen and heat, thereby preventing the pyrolysis and combustion of the wood. The char yield of the BCW group was 11.35%, whereas the char yield of the specimens treated with flame retardants was all above 20%, with the BO-1:APP-1 group reaching a char yield of 37.44%, which is a 230% increase compared to the BCW group.

CONCLUSIONS

This paper prepares *Pinus sylvestris* var. mongolicawood treated with combined flame retardants using a vacuum-pressure impregnation process and investigates the influence of the proportion of the combined flame retardants on the microstructure, mechanical properties, and flame retardancy of the wood. Based on the experimental results and analysis, the following conclusions can be drawn: *(1)* A uniform dispersion of the flame retardants in the wood pores was achieved, resulting in an excellent impregnation effect, which reduced experimental errors for subsequent experiments. *(2)* The weight gain rates of the flame-retardant-treated wood was around 10%, with a density greater than 0.6 cm^3/g, and the combined impregnation effect was superior to single-agent impregnation. Among them, the BO-1:APP-1 treatment had the best impregnation effect, with a weight gain rates reaching 10.36% and the highest density at 0.632 cm^3/g. *(3)* Apart from the APP single impregnation treatment, the other impregnation treatments improved the mechanical properties of Mongolian pine to some extent. The MOE and MOR of specimens treated with BO-1:APP-1 reached 12076 MPa and 116.3 MPa, respectively, which is an increase of 26.5% and 16.7% compared to the untreated MOE and MOR. *(4)* The oxygen index of the BCW group was 23.1%, while the oxygen index of the flame-retardant-treated specimens was all above 30%, and after the combined treatment, the oxygen index of the specimens was all above 35%. The BO-1:APP-1 group had the highest oxygen index at 42.5%, which is an 84% increase compared to the BCW group. The BCW group began to pyrolyze at 300°C, whereas the flame-retardant-treated specimens started to pyrolyze around 250°C, indicating that the pyrolysis stage of the flame-retardant-treated specimens was advanced by 50°C compared to the BCW group. The char yield of the BCW group was 11.35%, while the char yield of the flame-retardant-treated specimens was all above 20%, with the BO-1:APP-1 group reaching a char yield of 37.44%, which is a 230% increase compared to the BCW group.

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