Flame Retardant Properties of Laminated Bamboo Lumber Treated with Monoammonium Phosphate (MAP) and Boric acid/Borax (SBX) Compounds

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This study aimed to improve the flame-retardant properties of laminated bamboo lumber (LBL) using phosphorus-nitrogen-boron flame retardants (FRs). The combination of a 7:3 ratio of monoammonium phosphate (MAP) and boric acid/borax compounds (SBX), and 74.32 kg/m³ of FRs (10.3% weight gain), exhibited enhanced fireproofing performance for LBL materials. A commercial flame retardant (guanylurea phosphate) (GUP) was systematically studied as a comparison. A cone calorimeter and a thermal analyzer were used to characterize the combustion behavior and thermal stability, respectively. The flame retardants morphology in bamboo cell cavities was investigated using scanning electron microscopy (SEM) and an energy dispersive X-ray analysis (EDXA). The results showed that at a heat flux of 50 kW/m², the heat release rate and the total heat release of LBL samples treated with MAP-SBX flame retardants decreased more considerably than that of the untreated samples. The use of MAP-SBX not only promoted carbonization of LBL greatly but also indicated a good performance of smoke and combustion suppression as well as for the GUP. Flame retardants were confirmed to penetrate into the cell cavities of the bamboo using SEM and EDXA.

Keywords: Compounded flame retardants; Laminated bamboo lumber; Cone calorimeter; Fire-retardant behavior

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INTRODUCTION

Bamboo refers to a group of woody plants that belong to the grass Poaceae family and Bambusoideae subfamily (Ni *et al.* 2016). Due to the decline of wood resource availability and the increase of resource demand in the modern industrialized world, bamboo has gained popularity in the green building community. Bamboo has environmentally beneficial characteristics that are similar to wood: highly renewable, fast growth, biodegradable, sequesters carbon from atmosphere, low in embodied energy, and it creates less pollution during production than steel or concrete (Rittironk and Elnieiri 2008; Nath *et al.* 2009). Compared with other common building materials, bamboo is stronger than timber, and its strength-to-weight ratio is greater than that of common wood, cast iron, aluminium alloy, and structural steel (Mahdavi *et al.* 2011).

Although bamboo is a promising wood substitute, its hollow structure and small diameter limit its application where flat surfaces are required (Mahdavi *et al.* 2011; Li *et al.* 2013). To improve the utilization rate of bamboo and to enhance dimensional consistency, strength, and uniformity, bamboo can be split into small elements, disassembled into thin flat laminae, and then laminated together with adhesive to form various sizes and cross-sections of structural members (Sumardi *et al.* 2007; Corradi *et al.* 2009; Huang *et al.* 2013; Chen *et al.* 2013; Dixon *et al.* 2017). Laminated bamboo lumber

(LBL) is a composite lumber made from bamboo, which retains the high strength and toughness properties of bamboo and can be manufactured in designed dimensions similar to commercially available wood products (Rittironk and Elnieiri 2008; Sulastiningsih and Nurwati 2009).

The LBL not only shows excellent mechanical properties, but it also keeps the highly renewable characteristics of bamboo, which are the qualities of an ideal bamboo product. However, the drawbacks of the product are the flammable properties of its composition of cellulose, lignin, and hemicelluloses (Liu *et al.* 2012). When a fire occurs, the LBL materials will burn vigorously and increase the fire load, which can lead to casualties and economic losses. Therefore, the LBL must be treated with flame retardants before being used in the construction of structures or in decoration materials. At present, research of flame retardants for bamboo is mainly based on that of wood fire retardants. The application of phosphorus-nitrogen-boron synergistic flame retardants in the field of wood has matured into a relatively stable phase (Branca and di Blasi 2007; Chou *et al.* 2010; Zhao and Yu 2016). This provides beneficial theoretical support for the study of bamboo flame retardants.

The mechanism of the phosphorus-nitrogen synergistic flame retardant is generally considered the combination of the condensed phase and gas phase. When the materials burn, the flame retardant decomposes into phosphoric acid and poly-phosphoric acid, which form a non-volatile layer of protective film on the surface of the materials, preventing the oxygen from feeding the flame. At the same time, flame retardants can potentially release ammonia, nitrogen, water vapor, and other non-flammable gases that dilute the combustible gas and reduce the surface temperature of the materials by absorbing heat, which in turn prevents the burning of materials.

Boride is an inorganic fire retardant that increases dehydration reactions and reduces the amount of volatile organic compounds. Boric acid/borax is non-toxic to humans and animals, and is not only a flame retardant, but can also be used as an insecticide (Wang *et al.* 2004; Baysal *et al.* 2007). Previous studies have reported that the multi-component complex of flame retardants usually performs better than a single flame retardant element, due to the different mechanisms between each component (Ozcifci *et al.* 2007). The multi-component complex of flame retardants has become more mainstream in the modern world, such as the American Wood Protection Association (AWPA) FR-2 flame retardants, Dricon flame retardants, and GUP-boric acid mixture (FRW) flame retardants.

At present, there are many studies on bamboo flame retardants, but there are few published results on the flame-retardant behavior of LBL. The goal of the present work was to determine the flame performance of the LBL treated with a compound flame retardant. Boric acid/borax, and monoammonium phosphate compounds in appropriate proportions are utilized as the flame retardant to impregnate bamboo strips and then glue them together to form the LBL. The combustion performance, thermal properties and the FRs distribution in the bamboo's inner surface of the LBL were investigated.

EXPERIMENTAL

Materials

Dimension bamboo strips

Four-year-old moso bamboo (*Phyllostachys pubescens* Mazel ex H. de Lebaie) culms were obtained from Xinchang, in the Zhejiang province of China. The moisture content of the culms was conditioned between 8% and 10% in a kiln. Samples of the bamboo strips with dimensions 2100 mm \times 20 mm \times 5.5 mm (length \times width \times thickness) were manufactured in a series of processes, such as splitting, planning, and sawing. The density of the bamboo strips was approximately 0.75 g/cm³ to 0.80 g/cm³.

Phenol formaldehyde

Phenol formaldehyde (PF) resin was purchased from Beijing Dynea Chemical Industry Co. Ltd. (Beijing, China). The solid content of PF resin is 47.5%, with the following performance index values: a pH of 10.5, and a viscosity (20 °C) of 20 cps to 40 cps.

Flame retardant

The main components of the flame retardant (MAP-SBX) were purchased from Jinan Chemical Co. Ltd. (Jinan, China), which is a mixture of monoammonium phosphate $(NH_4H_2PO_4)$ and boric acid (H_3BO_3) /borax $(Na_2B_4O_7 \cdot 10H_2O)$ in a mass ratio of 7:3, the mixture of boric acid/borax (SBX) was composed of boric acid and borax in a mass ratio of 1:1. A commercial flame retardant (GUP), which mainly consists of guanidine polyphosphate and boride, was purchased from Guangzhou Beilin functional materials Co. Ltd. (Guangzhou, China) as a comparison. All of the agents used are commercially available industrial grade.

Methods

Retardancy treatment of bamboo strips

Aqueous solutions of the flame retardants were dissolved in water to a mass concentration of 20 % at room temperature. A 1% solution of triethanolamine ($C_6H_{15}O_3N$) was added into the flame retardant liquid to enhance the permeation of the flame retardant liquid into the bamboo strips.

The flame retardant was impregnated into the bamboo strips using the vacuumpressure method. The mass of the bamboo strips was measured before the impregnation. A vacuum of 0.08 MPa was applied and maintained for 30 minutes, then the flame retardant liquid was inhaled into the tank during the vacuum relief process. Afterwards, a constant pressure of 1.0 MPa was applied to the treatment tank for 2 hours. After the treatment, the bamboo samples were drained and weighted. Under the same saturation process, the weight percentage gain (WPG) of the control, MAP-SBX- and GUP-treated specimens, were 0%, 10.3%, and 10.1%, which were the same as the retention contents of 0 kg/m³, 74.32 kg/m³, and 73.61 kg/m³, respectively. The final moisture content of the bamboo samples was readjusted to 8% before performing the properties tests.

The weight percentage gain (WPG) (%, w/w) was calculated according to Eq. 1,

WPG (%) =
$$[(W_t - W_u) \cdot N / W_u] \times 100$$
 (1)

where W_u and W_t are the weights (g) of each bamboo samples before and after treatment, respectively, and N is the quality fraction of the flame retardant aqueous solution.

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Preparation of the flame retardant LBL samples

The process of producing the LBL began with the treated bamboo strips being ovendried at 60 °C for 8 h. After immersed in PF for 2 h and arranged in the longitudinal direction, the layered mats were placed at room temperature for 20 min. The layered mats were hot pressed on a pressing machine (Guosen Machinery Co. Ltd., Qingdao, China) with a hot press pressure of 3 MPa, a pressing time of 20 min, at a temperature of 130 °C. Figure 1 illustrates the key process used to manufacture the LBL products.



Fig. 1. The key process used to produce LBL products; a: bamboo tube; b: bamboo strips; and c: LBL samples

Cone calorimetric analysis

A cone calorimeter analysis is an effective approach used to evaluate the combustion behavior of flame-retarded materials. According to ISO 5660-1 (2002), the combustion performance of the LBL samples was tested using a cone calorimeter (FTT0242, Fire Testing Technology Co. Ltd., West Sussex, UK). The treated and untreated LBL samples were sliced into the dimensions of 100 mm \times 100 mm \times 19 mm (length \times width \times thickness). All tests were conducted at an irradiance level of 50 kW/m², which corresponded to a heater temperature of 800 °C. All specimens were tested in a horizontal orientation. The specimens were placed in the specimen holder and covered by the retainer frame. The test time was 1200 s to 1400 s. The heat release rate (HRR, kW/m²), total heat release (THR, MJ/m²), mass (%), mass loss rate (MLR, g/s), total smoke production (TSP, m²), specific extinction area (SEA, m²/kg), generation rate of carbon monoxide (CO, %), and CO yield (COY, kg/kg) were recorded and investigated.

Thermal properties

Thermogravimetric analysis was with a TGA analyzer (Q50, TA Instruments, New Castle, USA) at a heating rate of 10 °C/min in a N₂ atmosphere from room temperature to 800 °C. Approximately 7 mg to 10 mg of each sample was analyzed during the test.

Microscopy and microanalysis testing

Scanning electron microscopy (SEM) was performed by means of an ESEM instrument (FEG-XL30, FEI Company, Hillsboro, USA). The samples were covered with a galvanic platinum deposit with a current of 8 mA for 90 s by a sputter coater (Leica EM SCD005, Wetzlar, Germany). The acceleration voltage was 15 kV. Bamboo samples of approximately $5 \times 5 \times 1$ mm³ were sliced from the core of the LBL samples after the cone

calorimeter test. The microanalysis of chemicals in the cell cavity surface of the MAP-SBX-treated samples was determined by an energy-dispersive X-ray analysis (EDXA) (Hitachi S4800, Tokyo, Japan).

RESULTS AND DISCUSSION

Evaluations of Cone Test Results

Analysis of HRR and THR

The cone calorimeter based on the oxygen consumption principle has been widely used to evaluate the combustion characteristics of materials (Wu *et al.* 2010). The HRR and the THR were measured by the cone calorimeter, and are important parameters as they express the intensity of a fire (Fang *et al.* 2013), where a high flame retardant shows a low HRR peak value. Figure 2a illustrates the HRR curves, while Table 1 displays the key results of the cone calorimeter test (where pk-HRR₁ and pk-HRR₂, refer to the 1st and 2nd peaks of heat release rate, respectively).



Fig. 2. Graphs of the heat release with time for the control specimens and the flame-retardant-treated specimens; (a) HRR curves and (b) THR curves

Specimen Type	Peak Values of HRR (kW/m ²)		THR	Average MLR	Mass	TSP	Average SEA	COY ×100
	pk-HRR₁	pk-HRR₂	(MJ/m ²)	(g/s)	(%)	(m²)	(m²/kg)	(kg/kg)
Control	291.41(45)	381.97(730)	164.73	0.18	26.14	5.42	54.36	4.51
MAP-SBX	245.05 (40)	123.76 (925)	74.15	0.10	40.91	0.24	14.63	2.40
GUP	152.80 (50)	145.18 (1030)	80.10	0.11	37.40	0.12	12.25	2.51

Table 1. Cone Calorimeter	Test Results for Untreated	and Treated LBL Samples
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Note: The values in the brackets are the time when the peak appeared, unit is second

All the untreated and treated specimens showed two sharp HRR peaks, which is due to a special characteristic of thick char-forming substances found in wood materials (Schartel and Hull 2007). The HRR peak of the untreated specimens were sharper than the treated specimens. When the LBL specimen was ignited, combustible gas was emitted during decomposition, which transported a large quantity of heat and light at a high temperature, forming the first exothermic peak at approximately 45 s. The char then built up a protection layer on the surface of the LBL specimens until further decomposition occurred. Then the char layer cracked and reacted with oxygen to form the second exothermic peak at approximately 730 s. It has been shown that the HRR is the most important property of fire hazards because it indicates the intensity of a fire (Babrauskas and Peacock 1992). The second HRR peak is more critical than the first one for wood materials (Liu *et al.* 2012).

The pk-HRR₂ of the MAP-SBX and GUP specimens were 123.76 kW/m² and 145.18 kW/m², which decreased 67.60% and 61.99%, respectively, and separately compared to the untreated specimens. Their corresponding appearing times were delayed by 195 s and 300 s, which inhibited the final deflagration and exothermic reaction of the materials that can be vital for getting people to escape from a fire. As indicated by the reductions in heat release rates, the MAP-SBX showed a better performance in reducing the pk-HRR₂ of the LBL specimen compared to the GUP, which was considered dominant in the whole heat release process.

The mechanism of the flame retardants is that phosphorus and nitrogen elements become pyrolyzed into phosphoric acid and metaphosphoric acid at high temperatures with strong dehydration and carbonization. When the boride decomposes it absorbs a large amount of heat with the help of water loss at a high temperature, so the surface temperature of the materials is reduced (Wang *et al.* 2004). At the same time, boron oxide from the pyrolysis products forms a viscous coating that covers the surface of the materials, which acts a barrier against oxygen and heat (Tomak *et al.* 2012).

As shown in Table 1 and Fig. 2b, it is worth noting that the THR of the treated samples remarkably decreased. The THR of the GUP specimen decreased 51.37% compared to the untreated specimen. Meanwhile, the MAP-SBX shows equal flame retardant efficiency, which decreased the THR of the LBL specimen from 164.73 MJ/m^2 to 74.15 MJ/m^2 by 54.99%. These two kinds of flame retardants reduced the fire damage significantly, and have the same excellent fire protection effect.

Analysis of MLR and Mass

The mass loss rate (MLR) indicates that the sample mass changes as a function of time during combustion (Huang *et al.* 2010). It also reflects the thermal stability of the sample. The MLR of the LBL samples are shown in Table 1 and Fig. 3a. The variation

trends of the LBL's MLR curves were consistent with their HRR curves, which was evidence that the mass loss and heat released during combustion were simultaneous. In the early stages of combustion, the carbon layer of the LBL specimens had not yet been formed, so there was a higher rate of mass loss that corresponded to the first peak on the MLR curve. In the late combustion stage, the LBL specimen showed a dramatic mass loss because of the decomposition and rupture of the remaining material, which corresponded to the second peak of the curve on the MLR.



Fig. 3. Graphs of the mass loss with time for the control specimens and the flame-retardant-treated specimens; (a): MLR curves; (b): Mass curves

As shown in Table 1 and Fig. 3b, the mass curves of all the specimens showed similar changes. In the flaming phase, the mass of the residual material declined rapidly. The thermal stability of the LBL greatly was improved after being treated with the flame retardant, the slope of the curve became smaller, which corresponded to the decrease of the rate of pyrogenic decomposition. In the final stage, during which the calcination of carbon residue occurred, the curves of the mass turned almost horizontal. The residual mass of the untreated, MAP-SBX-, and GUP-treated specimens were 26.14%, 40.91%, and

37.40%, respectively. The char formation of the LBL was accelerated by the MAP-SBX and GUP flame retardants, which plays a vital role in reducing the mass loss rate (Leung *et al.* 1996).

Analysis of gas release

Research suggests that most fire victims die from the inhalation of excessive smoke (Chow 2003; Ma *et al.* 2012), which consists of a very large number of toxic components (Li *et al.* 2011; Prabhakar *et al.* 2015). Therefore, the TSP is also an important index of flame retardants. As shown in Table 1 and Fig. 4a, the final TSP of the untreated, MAP-SBX and GUP specimens were 5.42 m^2 , 0.24 m^2 , and 0.12 m^2 , respectively. It was found that the treated LBL had almost parallel TSP curves, which were much lower than the untreated LBL curves. The generation of N₂ and NH₃ gas diluted the concentration of oxygen as well as the flammable gases generated during the bamboo thermal degradation. Therefore, this slowed down the gas-phase oxidation rate and the bamboo thermal degradation rate, then the heat and smoke released during the LBL combustion during the combustion process, and the formation of a glassy film not only hindered the heat diffusion into the LBL, but it also delayed the evaporation of flammable gases to some extent, and then it inhibited the LBL combustion and smoke release.



Fig. 4. Graphs of the gas release with time for the control specimens and the flame-retardant-treated specimens; (a) TSP curves, (b) SEA curves, (c) COY curves, and (d) CO curves

The curves of the SEA (Fig. 4b) shows that the production of smoke primarily occurred during the later stages of combustion, and the tendency of the SEA was similar to that of the HRR. This was likely due to the carbon generated during the low temperature stages that oxidized and dissociated in the later stages at higher temperatures. One part of the heat was released from the thermal oxidation of the carbon, which was transmitted to the inner material, and the oxygen-deficient combustion of residual mass was then able to produce more flue gas (Zhang *et al.* 2016). The SEA peaks of treated LBL samples were lower than the untreated LBL samples, and the main flue gas had a delayed appearance, which substantially reduced smoke damage.

The generation of CO in a fire is very dangerous. Considering the suffocation risk, it could be the large toxic component that makes smoke inhalation deadly (Hirschler 2006). Figures 4c and 4d reflect the generation rate of carbon monoxide (CO) and the CO yield (COY). A two-stage release of CO was observed, and the generation rate of CO dropped remarkably after the flame retardant treatment. The average COYs of MAP-SBX and GUP specimens were reduced 46.7% and 44.4%, respectively compared to the untreated samples.

During the process of smoke production, non-combustible gas was released from the flame retardant in the initial ignition phase that diluted the surrounding oxygen. A large amount of carbon was also formed due to the catalytic effects of monoammonium phosphate. In the late phase of burning, a large amount of CO was produced due to the decomposition of the charred layers and the inadequate oxidation of charcoal. According to the above analysis of TSP, SEA, and COY, which were reduced by 95.6%, 73.1% and 46.7% after MAP-SBX treated, respectively, it can be concluded that MAP-SBX have favorable effects in smoke suppression as well as GUP.

Thermal Properties

Thermal analysis is a method to study the relationship between the physical properties of substances and temperature variation under the automatic control program of temperature, which serves as evaluation criteria for flame retardant properties (Ozawa 2000; Hu *et al.* 2000). Table 2 shows the initial decomposition temperatures (T_i), maximum degradation temperatures (T_m), and carbon residual rate of the untreated and treated LBL specimens. The T_i and T_m of all specimens decreased after the flame retardant treatment, which indicated that the flame retardants changed the pyrolysis process in bamboo.

	<i>T_i</i> (°C)	<i>T_m</i> (°C)	Carbon Residual Rate(%) at Different				
Specimens			Temperatures				
-			200 °C	400 °C	600 °C	800 °C	
Control	193.16	337.74	95.44	22.99	17.80	16.38	
MAP-SBX	171.59	315.84	93.71	43.08	30.97	27.92	
GUP	176.35	331.20	93.06	42.07	33.62	30.13	

Table 2. Thermal Analysis Parameters of Treated and Untreated Specimens

Figure 5a shows the pyrolysis process of bamboo at a heating rate of 10 °C/min. As shown on the TG curves, the untreated and treated samples underwent three degradation steps. The first degradation step of the mass loss was in the temperature range of 30 °C to 245 °C, which is due to the removal of water from the samples (Liu *et al.* 2013). The mass loss of the treated samples at 200 °C were greater than 5%, which was due to the catalytic dehydration of monoammonium phosphate. The second degradation step was observed in

all samples at approximately 245 °C to 360 °C, the degradation of cellulose, hemicellulose, and lignin account for the weight loss during this stage (Brebu and Vasile 2010; Wang *et al.* 2013). Compared to the untreated samples, the MAP-SBX samples began to degrade at 171.6 °C and the critical temperature of the maximum mass loss was 315.8 °C, which was approximately 21.6 °C to 21.9 °C lower than the untreated, respectively.



Fig. 5. TG curves of treated and untreated LBL; (a) TGA curves and (b) DTG curves

The function of the flame retardant in wood materials is to increase the char at the cost of flammable volatile products (Bagga *et al.* 1990). As with the DTG curves (Fig. 5b), it was found that the width of the second peaks of the weight loss rate were narrowed. The decrease of the thermal decomposition scope implied that the thermal stability of the materials was improved with the addition of the flame retardants. The last degradation stage was the calcination of the remaining materials from 360 °C to the end of the reaction. The residues of the MAP-SBX and GUP samples detected at 800 °C were 27.9% and 30.1%, respectively, which exceeded that of the untreated sample (16.4%). The increase of residual mass after the flame retardant treatment revealed that the addition of flame retardants facilitated the formation of more char at the expense of flammable volatile products of thermal degradation, which is conducive to the inhibition of the combustion process (Gao *et al.* 2006).

Morphology Analysis and SEM Analysis

The study of char morphology is useful in explaining the differences between the untreated and the treated samples due to the important role that char plays in flame retardants' performance. Figures 6a, b, and c show the camera images of the LBL specimens after a cone calorimeter test. It was quite clear that the untreated LBL sample burned into ashes with less residue. By contrast, the LBL specimens treated with flame retardant demonstrated a condensed and smooth carbon layer on their surfaces with a great quantity of rigid carbon blocks, which contributed to a higher flame retardant efficiency.

Figures 6d through g are the SEM images of the char residue after a cone test, it was observed that the cell cavity surface of the untreated bamboo samples was relatively smooth without any flame retardant residue. However, a dense molten coating covered the surface of the bamboo cell cavity after treatment with the flame retardant, which inhibited the release of combustible gas and smoke, isolated oxygen from outside, and prevented heat transferring.



Fig. 6. Camera images and SEM micrographs of untreated and treated LBL samples after cone calorimeter test; (a) untreated sample; (b) MAP-SBX sample; (c) GUP sample; (d) MAP-SBX 150x; (e) untreated 4000x; (f) GUP 5000x; (g) MAP-SBX 5000x; (h) EDAX spectra of MAP-SBX treated LBL sample

The EDXA spectrum (Fig. 6h) shows the element composition of a small area on the cell cavity coating of the MAP-SBX-treated LBL samples after a cone calorimeter test, marked by a red arrow (Fig. 6g). The results indicated that N, P, and B were present in the cell cavity surface of bamboo, which suggested that the MAP-SBX flame retardant had penetrated the cell cavity of bamboo after the vacuum pressure impregnation treatment. This played an important role in the flame retardant capabilities and smoke suppression during the burning of the LBL samples.

CONCLUSIONS

- 1. Some properties of laminated bamboo lumber (LBL), manufactured from bamboo strips treated with flame retardant, were investigated. The LBL that had been treated with monoammonium phosphate (MAP) - boric acid/borax compounds (SBX) possessed a high fire resistance, equal to that of the commercial flame retardant guanylurea phosphate (GUP), when used for the LBL materials. Its preparation method was simple and showed good solubility. When the LBL samples after being treated with MAP-SBX were compared with the untreated samples, the second heat release rate (pk-HRR₂), and the total heat release (THR) dropped in value by 67.6% and 55.0%. The total smoke production (TSP), as well as, the CO yield (COY), were decreased 95.6% and 46.7%, respectively. Monoammonium phosphate and boride compounds exhibited a good effect of catalytic charring, which was embodied in the formation of a carbon layer on the cone calorimeter samples, and a large increase of residual weights by 56.5% and 70.5% for Mass (cone calorimeter test) and residue (TG test). The EDXA spectra of the MAP-SBX-treated LBL samples after a cone calorimeter test confirmed that N, P, and B were evenly present, and formed a dense molten coating that covered the cell cavity surface of the bamboo.
- 2. In accordance with the congruent relationship between the cone calorimeter test and the Chinese national standard GB 8624 (2006), the LBL materials treated with MAP-SBX met the level B of flame retardant materials. In conclusion, after the flame retardant treatment, the LBL materials were suitable for building and decoration materials (wall, furniture and flooring).

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