



Evaluating the Flame Retardancy of Shaving Super Bamboo Layer by Layer Self-Assembly With Phytic Acid-Polyethyleneimine

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To improve the flame retardancy of bamboo materials, layer by layer (LbL) self-assembly of phytic acid (PA)-polyethyleneimine (PEI) on the surface of shaving super bamboo specimens with different solution concentrations of PA-PEI and times of LbL self-assembly was completed in this study. Fourier transform infrared analysis results showed that PEI was well assembled to the surface of bamboo specimens by a hydrogen bond with PA as intermediation. The application of PA and PEI significantly promoted the formation of carbon residue, as characterized by simultaneous thermal measurements. Particularly, the peak heat release rate and total heat release rate of bamboo self-assembly with 10 wt% PA and 10 wt% of PEI solution were reduced by 19.36 and 22.3%, respectively. The treated bamboo specimen showed increases of 35.56 and 480.70% in fire performance index and residual mass, respectively, compared to the control sample. Besides, yields of CO and CO₂ were decreased by 17.77 and 17.07% in comparison with the control group, respectively. The LbL self-assembly with PA-PEI can effectively improve the flame retardancy of bamboo materials by promoting the formation of a residual char layer.

Keywords: shaving super bamboo, phytic acid (PA), polyethyleneimine (PEI), self-assembly, flame retardancy

INTRODUCTION

Bamboo presents high application prospects and economic value as a substitute for wood due to its similarity to wood in compositions and structures (Scurlock et al., 2000; Zhang X. et al., 2019; Zhang L. et al., 2020). It is traditionally applied to fabricate bamboo-based panels such as plywood, fiberboard, laminated bamboo board, and particleboard. All of these bamboo-based panels have always maintained an important position in the manufacturing of flooring, furniture, and construction due to their unique excellent characteristics, but their high flammability has significantly limited various applications. Therefore, it is necessary to develop flame retardant bamboo-based panels for their practical applications (Guo et al., 2019; Pope et al., 2019).

Most of the previous studies on the preparation of flame retardants focus on green and sustainable flame retardants (He et al., 2020). As a natural non-toxic compound, phytic acid (PA) is abundant in beans, cereals, and oilseeds (Dusková et al., 2001; Fang et al., 2019; Liu et al., 2021). As a biocompatible, environmentally friendly, non-toxic, and easily available organic acid, the content

of phosphorus reaches 28%. The phosphorus element makes it a potential green biomass flame retardant. Negative charges were found in the PA solution due to the existence of the special structure that 12 acidic groups were symmetrically connected to a cyclohexanol ring (Jo et al., 2008; Ye et al., 2012; Zeng et al., 2021). The thermal stability and mechanical property of the matrix were declined once combined with PA. It has been reported that the combination of PA and certain metal ions can significantly improve the flame retardant performance of PA (Shen et al., 2016; Fang et al., 2018; Gong et al., 2020). A synergistic effect is observed to generate phosphorus-containing flame retardants with the presence of nitrogen elements, leading to a more excellent flame retardant performance (Nguyen et al., 2013). Therefore, PA is more often combined with some nitrogen-containing compounds such as chitosan, melamine, and polyethyleneimine because the combination results in a synergistic fire-retarding effect once they are used as a flame retardant (Zhang et al., 2014a; Zhang et al., 2014b; Jin et al., 2017; Wang et al., 2019; Zmz et al., 2019). It has been proved that the combination of PA with nitrogen-containing compound will be made into an intumescent flame retardant (IFR) system with PA as an acid source and phosphorus source (Dusková et al., 2001; Zhang et al., 2014a; Jin et al., 2017; Zeng et al., 2021). In addition, a gas source in the IFR system is provided by nitrogen-containing compounds with releasing incombustible gases of N_2 and NO_2 in the combustion process of composites (Nguyen et al., 2013; Zhang et al., 2014b; Xiong et al., 2019). This flame retardant system is usually evenly dispersed in high molecular polymers to exert a significant flame retardant effect. It has been found that PA is effectively absorbed by the wool fabric and excellent flame retardant performance has been illustrated with the electrostatic interactions between the positively charged amino groups and the negatively charged phytic acid phosphate groups in wool (Cheng et al., 2016a; Cheng et al., 2019).

PA can be effectively combined with cellulose-rich materials to prevent cellulose-rich materials from combusting during the combustion process of materials due to the action of the condensed phase mechanism (Wang et al., 2015; Zhang Z. et al., 2019). However, a further treatment of PA is needed to get a neutral negative charge carried by PA and to make it effectively adhere to the surface of the matrix. Some efforts have been made to prepare an intumescent flame retardant (IFR) solution with PA and uracil to improve the flame retardant performance of the matrix. The IFR solution with PA and uracil has been used to prepare one-step treated wood for excellent flame retardancy. Results revealed that the peak heat release rate, total heat release, smoke production rate, and total smoke production of flame retardant wood were reduced by 41, 30, 61, and 56%, respectively, compared with natural wood (Zhang Y. et al., 2020).

The IFR system completed by the LbL self-assembly technology has been preliminarily introduced to surface modification treatment of shaving super bamboo in this study. PA is selected to provide an acid source and phosphorus source to improve the flame retardant

performance, and polyethyleneimine (PEI) provides a gas source for its high nitrogen content and positive charge in solutions (Liu et al., 2016; Ran et al., 2019; Huo et al., 2021). Besides, an LbL self-assembly method was applied to assemble and install PA to be negatively charged and PEI to be positively charged on the surface of shaving super bamboo to fabricate bamboo materials with excellent flame retardant performance (Laachachi et al., 2011; Laufer et al., 2012; Wang et al., 2017; Fang et al., 2019). Herein, we systematically analyze the effects of PA-PEI solution and times of LbL self-assembly on the flame retardancy of bamboo specimens. Besides, PA-PEI LbL self-assembly on the surface of bamboo specimens was analyzed by Fourier transform infrared (FTIR) analysis and simultaneous thermal analysis (STA). Cone calorimeter analysis was completed to investigate further the effect of reagent concentration and times of LbL self-assembly on flame retardant of bamboo specimens LbL self-assembly with PA-PEI. This work provides an insight to a better understanding of the flame retardancy of shaving super bamboo LbL self-assembly with PA-PEI.

EXPERIMENTAL SECTION

Materials

Shaving super bamboo with demotions of 2000 mm (length: L) \times 500 mm (width: W) \times 0.6 mm (thickness: T) was obtained from local Bamboo Industry Co., Ltd. Phytic acid (PA, $C_6H_{18}O_{24}P_6$, concentration: 50 wt%) with a component of 660 and polyethyleneimine (PEI, CH_2CH_2NH , AR, concentration: 99 wt %) with a component of 600 were purchased from Aladdin Biochemical Reagent Co., Ltd., Shanghai, China.

Layer by Layer Self-Assembly of Bamboo Specimens With Phytic Acid-Polyethyleneimine

Shaving super bamboo was sawn into chips with the dimension of 100 mm (L) \times 100 mm (W) \times 0.6 mm (T). All of the bamboo chips were evenly divided into three groups with 100 specimens in every group, namely, Group A, Group B, and the control group. The technology of LbL self-assembly with PA-PEI solution was first applied to improve the flame retardancy performance of shaving super bamboo. The PA solution was prepared by mixing PA with deionized water by magnetic stirrers (DF-101S, Shanghai Lichen Instrument Technology Co., Ltd., Shanghai, China) for 20 min to obtain a homogenized solution at room temperature. The PEI solution was prepared in the same method. Diagrammatic representation of bamboo specimens LbL self-assembly with PA-PEI is illustrated in **Figure 1**. Bamboo chips in Groups A and B were evenly divided into five groups and marked as A1 to A5 and B1 to B5, respectively. Before LbL self-assembled with PA-PEI, all of the bamboo chips were immersed in water at 70°C for 15 min and dried at $60 \pm 1^\circ C$ in a drying oven (DKN611, Yamato Scientific Co., Ltd. Tokyo, Japan) for 30 min and then at $103 \pm 2^\circ C$ until the constant mass is reached when two successive weights do not differ by more than 0.1%. The bamboo specimens

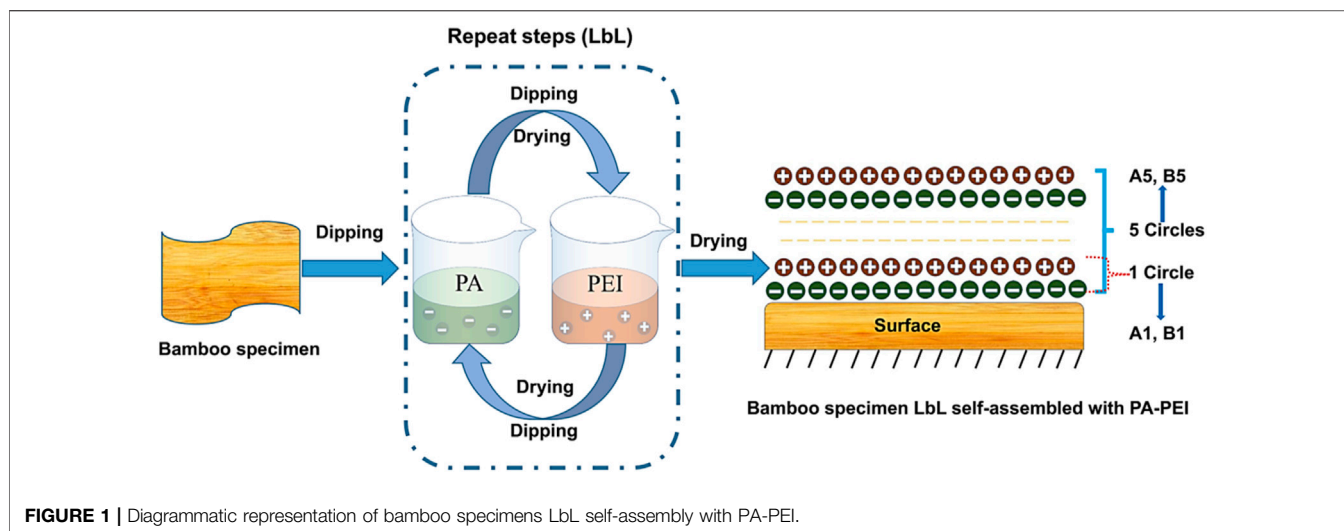


FIGURE 1 | Diagrammatic representation of bamboo specimens LbL self-assembly with PA-PEI.

TABLE 1 | Bamboo specimens with different solution concentrations and times of LbL self-assembly with PA-PEI.

Sample no.	PA concentration (%)	PEI concentration (%)	Times of LbL self-assembly
The control	0	0	0
A1	5	5	1
A5	5	5	5
B1	10	10	1
B5	10	10	5

with different solution concentrations and times of LbL self-assembly with PA-PEI are shown in **Table 1**. Bamboo chips in Group A1 were immersed in 5 wt% PA solution for 5 min and then dried at $60 \pm 1^\circ\text{C}$ until the moisture content (MC) reduced to 12%. Besides, the bamboo chips were dipped in a 5 wt% PEI solution for 5 min and dried at $60 \pm 1^\circ\text{C}$ for 30 min and then at $103 \pm 2^\circ\text{C}$ until the constant mass is reached. Bamboo specimens in Group A5 were obtained by repeating the preceding steps five times. Bamboo specimens in Group B were LbL self-assembled by immersing in the 10 wt% PA solution and 10 wt% PEI solution using the same processing step as Group A. Besides, bamboo specimens in Group B5 were immersed in the 10 wt% PA solution and 10 wt% PEI solution five times.

Weight Percent Gain of Bamboo Specimens Self-Assembly With Phytic Acid-Polyethyleneimine

Weight percent gain (WPG) of bamboo specimens LbL self-assembly with PA-PEI was calculated according to **Equation 1**:

$$WPG = \frac{M_2 - M_1}{M_1} \times 100\% \quad (1)$$

where M_1 is the oven-dry weight of bamboo samples before LbL self-assembly treatment in g and M_2 is the oven-dry

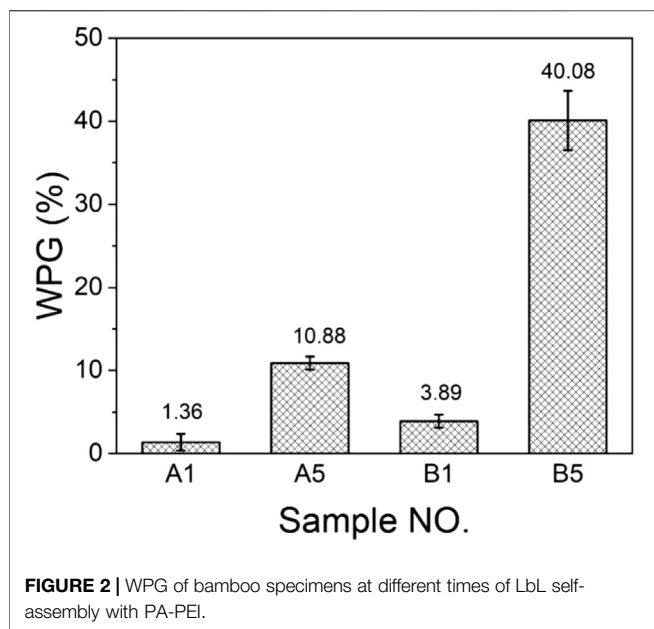
weight of bamboo samples after LbL self-assembly treatment in g.

Fourier Transform Infrared Analysis of Bamboo Specimens Layer by Layer Self-Assembly With Phytic Acid-Polyethyleneimine

FTIR analysis of bamboo specimens before and after LbL self-assembly with PA-PEI was completed by spectroscopy (Nicolet 6700, Nicolet Co., Ltd., United States). The investigation was conducted in the $400\text{--}4,000\text{ cm}^{-1}$ frequency range (Zhang X. et al., 2019). The tested specimen for FTIR test was produced by mixing 1 mg bamboo powders (i.e., oven-dry state) with a size of sieve opening ranges from 80 to 120 with 99 mg potassium bromide (KBr).

TG-DTA Analysis of Bamboo Specimens Layer by Layer Self-Assembly With Phytic Acid-Polyethyleneimine

TG-DTA analysis of bamboo specimens before and after LbL self-assembly with PA-PEI was completed by a simultaneous thermal analysis (STA 409 C, NETZSCH Company, German). Ten mg of bamboo powders was placed in an Al_2O_3 crucible and measured by heating from 20°C to 800°C with a heating rate of $10^\circ\text{C}/\text{min}$ in the nitrogen atmosphere.



Cone Calorimeter Analysis of Bamboo Specimens Layer by Layer Self-Assembly With Phytic Acid-Polyethyleneimine

The combustion performance of bamboo specimens was measured by a cone calorimeter (Fire Testing Technology Ltd., United Kingdom) in accordance with ISO 5660-1 (Yu et al., 2016; Hou et al., 2017). All specimens with dimensions of 100 mm (L) × 100 mm (W) × 6 mm (T) were hot-pressed at 100°C for 5 min through laminating shaving super bamboo specimens (100 mm (L) × 100 mm (W) × 0.6 mm (T)). Prior to cone calorimeter testing, the tested specimens were conditioned to equilibrium moisture content (EMC) at a controlled environment (Temperature of 23°C; 50% relative humidity (RH)) in a high-humidity alternating test box (EL-10KA, Espec Corporation, Hudsonville, MI, United States) until the constant mass is reached. The tested specimens were irradiated with an incident heat flux of 50 kW (Yu et al., 2016; Hou et al., 2017). All the experimental treatments were performed three times with the average values reported.

RESULTS AND DISCUSSION

Weight Percent Gain of Bamboo Specimens Self-Assembly With Phytic Acid-Polyethyleneimine

WPG of bamboo specimens before and after LbL self-assembly is illustrated in **Figure 2**. A gradual increase was observed in WPG with the increasing times of LbL self-assembly as illustrated in **Figure 2**. A slight increase of 1.36% was obtained to A₁ as compared with the untreated bamboo specimens. However, an increment of 10.88% was generated with the further increase times of LbL self-assembly to five of A₅ and over seven times than

that of A₁. It was also noted that WPG of B₁ and B₅ was 3.89 and 40.08%, respectively. An obvious increase was generated compared to that of A₁ (1.36%) and A₅ (10.88%), indicating that the increment was caused by increasing concentration of PA-PEI solution. Therefore, it was concluded that adsorption capacities of PA and PEI were increased with the treatment by LbL self-assembly method and the increase of solution concentration.

Fourier Transform Infrared Analysis

FTIR curves of untreated and treated bamboo specimens are presented in **Figure 3**. PA₅ and PA₁₀ in **Figure 3B** are bamboo specimens treated with 5 wt% and 10 wt% of PA solutions, respectively. That of PEI₅ and PEI₁₀ are bamboo specimens treated with 5 wt% and 10 wt% of PEI solutions, respectively. An obvious decrease in the peak intensity at 3,440 cm⁻¹ indicated the reduction of the content of free -OH in bamboo specimens after LbL self-assembly. Actually, PA was speedily assembled to the surface of bamboo specimens through physical absorption by hydrogen bond, indicating the excellent connection between PA and bamboo specimens (Guo et al., 2019). And then, PEI was also combined with PA by hydrogen bond during the immersion of bamboo specimens in PEI solutions. Hence, PEI was assembled to the surface of bamboo specimens with PA as intermediation. This is the reason why bamboo specimens were first dipped in PA solution and then PEI solution in our study. It was also noted that the band at approximately 1,633 cm⁻¹ of O-P-O (Zhang et al., 2014a) and P=O double bond stretching in the range of 1,200–1,260 cm⁻¹ (Jiang et al., 2012) originated from PA clearly appear as illustrated in **Figure 3**. Combination between free -OH in bamboo specimens and O-P-O in PA was speedily formed with the immersing of bamboo specimens in PA solution, further leading a reduction in both the peak intensity of free -OH in bamboo specimens and O-P-O in PA. Besides, the band at 1,495 cm⁻¹ was caused by symmetric and asymmetric bending vibration of -NH₂ in PEI (Wang et al., 2014). The functional group of -NH₂ in PEI was combined with P=O in PA with the immersion of bamboo specimens in PA solution. The absorption peak at 1736 cm⁻¹ corresponding to the C=O stretching in the carboxyl (Duygu et al., 2008) of bamboo specimens was also observed. Cellulose was evidenced to exist in the bamboo specimens with the presence of the peak intensity at 1,056 cm⁻¹ of C-O-C. A decrease in the peak intensity ratio between C-O-C and O-P-O indicated the depositing of PA on the surface of bamboo specimens. In summary, a thin film of PA-PEI was evidenced to be generated on the surface of treated bamboo specimens after LbL self-assembly with PA-PEI solution in this study.

TG-DTA Analysis

Figure 4 shows the TG and DTG curves of bamboo specimens before and after self-assembly with PA-PEI. The maximum weight loss rate (WLR_{max}) and the temperature (*t*) to WLR_{max} of bamboo specimens before and after self-assembly with PA-PEI are listed in **Table 2**. The pyrolysis process of bamboo is mainly divided into three stages in accordance with the heat treatment temperature (Zhang Y. et al., 2020). An obvious weight loss was observed in bamboo specimens before and after self-assembly

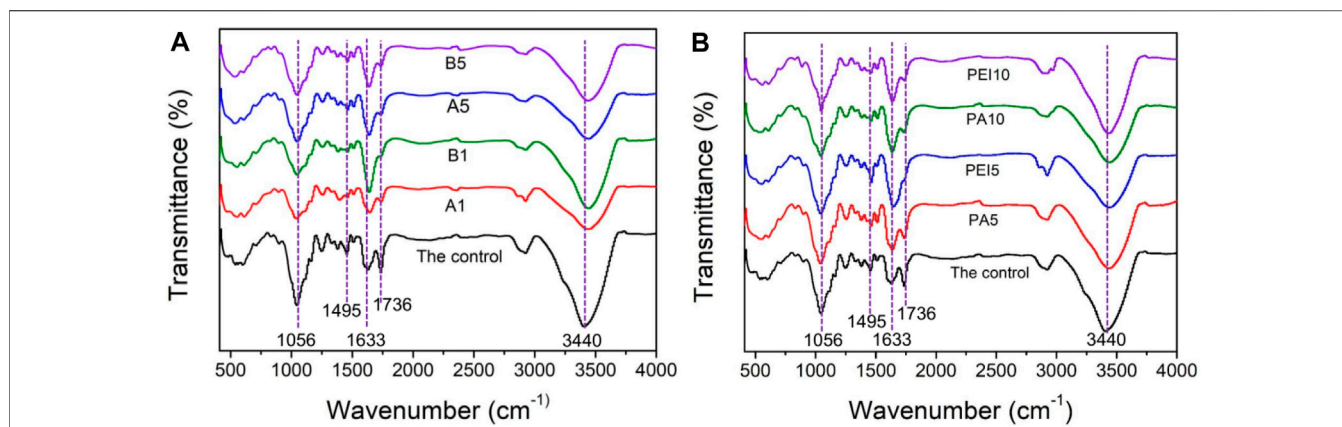


FIGURE 3 | FTIR curves of bamboo specimens: **(A)**, bamboo specimens before and after self-assembly with PA-PEI; **(B)**, bamboo specimens before and after treatment with PA solution and PEI solution.

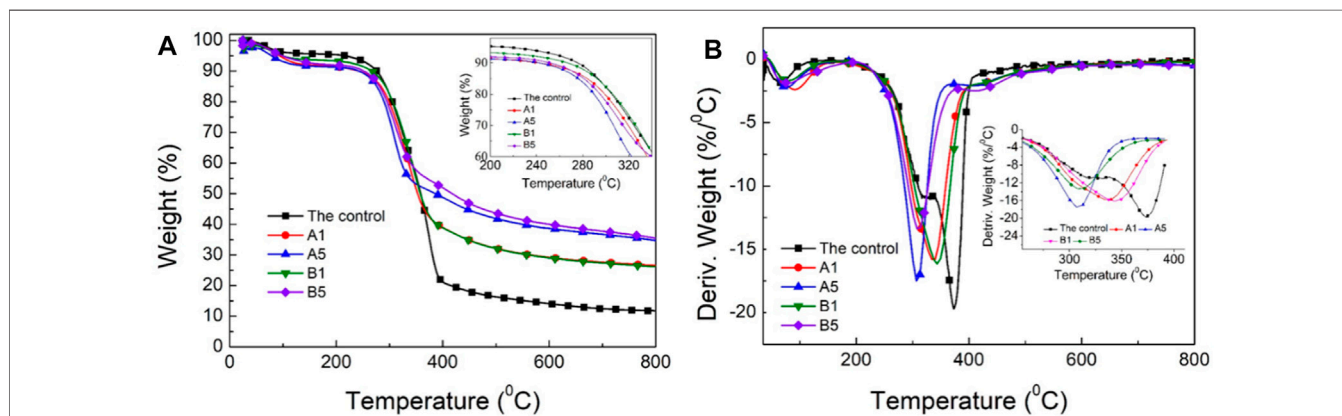


FIGURE 4 | Weight curves **(A)** and DTG curves **(B)** of bamboo specimens before and after self-assembly with PA-PEI in N₂ atmosphere.

TABLE 2 | WLR_{max} and *t* of bamboo specimens in the pyrolysis process.

Sample no.	1 st WLR _{max} (%/°C)	<i>t</i> ₁ (°C)	2 nd WLR _{max} (%/°C)	<i>t</i> ₂ (°C)	Residual mass at 800°C (%)
The control	4.40	66.93	19.69	373.13	11.73
A1	8.22	91.10	15.77	333.88	26.55
A5	8.67	75.89	17.49	306.81	34.76
B1	6.37	80.36	16.14	343.46	26.17
B5	7.88	80.34	13.35	307.9	35.51

with PA-PEI in the temperature ranging from 80 to 90°C in the first stage of the pyrolysis process as shown in **Figure 4B**. Compared with WLR_{max} of the control group (4.4%/°C), the WLR_{max} of treated bamboo specimens was obviously increased by 86.8% (A1) and 95.9% (B1), respectively. However, the corresponding increment of WLR_{max} was 44.8% (A5) and 79.1% (B5) with the further increase times of self-assembly to five. Besides, *t* of the treated bamboo specimens was also increased by 36.1% (A1), 13.4% (B1), 20.1% (A5), and

20.0% (B5), respectively. This may be due to the evaporation of moisture in bamboo specimens in accordance with the pyrolysis process of bamboo and wood (Duygu et al., 2008; Poletto et al., 2012). Besides, overflow of trace free organic molecules for PA was also observed in the pyrolysis process of the treated bamboo specimens according to the boiling points of PA and PEI. A slight difference between bamboo specimens before and after self-assembly with PA-PEI was observed due to the evaporation of PA.

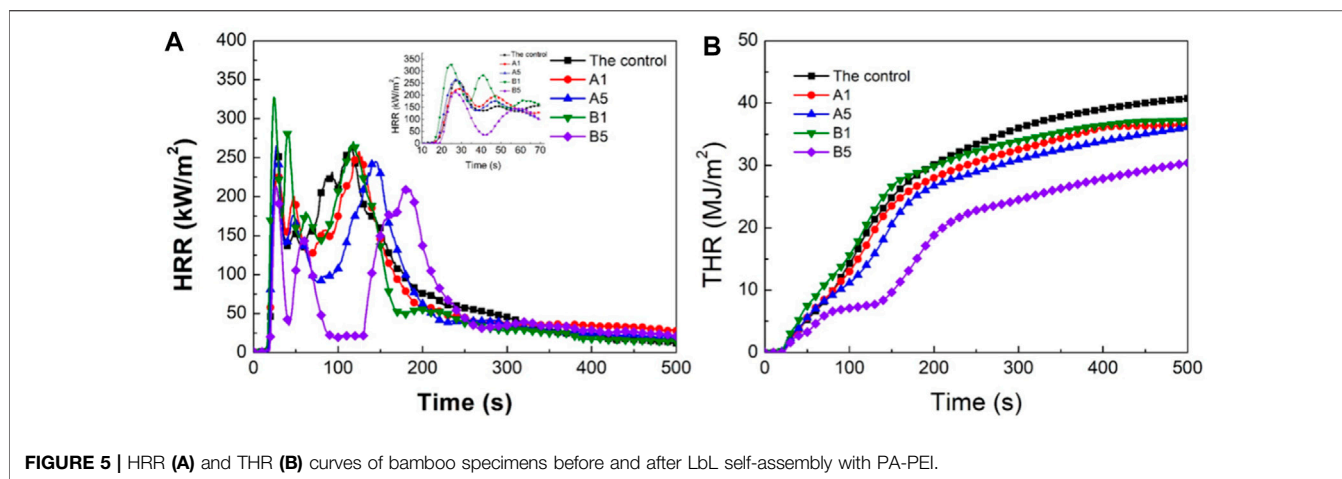


FIGURE 5 | HRR (A) and THR (B) curves of bamboo specimens before and after LbL self-assembly with PA-PEI.

As listed in **Table 2**, WLR_{max} of the treated bamboo specimens in the second stage of the pyrolysis process was decreased by 19.91% (A1), 11.17% (A5), 18.03% (B1), and 32.20% (B5) in comparison with that of the control group (19.69%/°C), respectively. Moreover, t of A1 (333.88°C) and B1 (343.46°C) were slightly decreased by 10.52 and 7.95%, respectively. The decrements of t were increased to 17.77% (A5) and 17.48% (B5) with the further increase time for LbL self-assembly with PA-PEI to five. In addition, it was found that residual mass at 800 °C was significantly increased to 126.34% (A1) and 123.10% (B1) after the first time of LbL self-assembly with PA-PEI. A significant increase in residual mass was generated with the increase of times for LbL self-assembly with PA-PEI to five, which was nearly doubled (i.e., A5: 196.33% and B5: 202.73%). This may be due to the PA-PEI thin film formed on the surface of treated bamboo specimens in the LbL assembly process with PA-PEI solutions. The thin film was thick enough to prevent bamboo specimens from thermal decomposition with the increase of concentration of PA-PEI solution and times of LbL self-assembly. Hence, a remarkable increase was found in the residual mass at 800°C of treated bamboo specimens (Shen et al., 2016).

It has been reported that the peak at 320 ~ 330°C and 373°C of bamboo material under N_2 atmosphere is the degradation of hemicellulose and lignin, respectively (Brebú and Vasile, 2010; Herrera et al., 2014). Additionally, incombustible gases, such as N_2 , are always generated in the decomposition process of PEI in the phosphorus-nitrogen flame retardant system (PA-PEI) formed on the surface of bamboo specimens after LbL self-assembly with PA-PEI and a relatively weak contribution to the TG-DTG curve in this study. The decomposition of PA was generated in a low-temperature stage, and metaphosphoric acid was formed in this stage with PA as an acid source. Besides, binding interaction between metaphosphoric acid and the matrix occurred and further resulted in the formation of a dehydration carbon layer. Moreover, the degradation chain reaction of the matrix shifts to the formation of the carbon layer and leads to the increase in the carbon residue contents (Jeng et al., 2002; Daneluti and Matos, 2013; Cheng et al., 2016b). Therefore, the maximum degradation rate of the substrate comes earlier at a lower

temperature. The same conclusion was also obtained by increasing residual mass, as shown in **Figure 4A**, **Table 2**, and the attenuation of the second peak in DTG curves illustrated in **Figure 4B**. It was also noted that times of LbL self-assembly with PA-PEI makes a great difference to pyrolysis process of bamboo specimens. For the bamboo specimens LbL self-assembly with PA-PEI, the following order of residual mass was found: B5>A5>B1>A1. Hence, the bamboo specimens LbL self-assembly with PA-PEI five times (A5 and B5) showed the highest residual mass.

Cone Calorimeter Analysis

Heat Release Rate and Total Heat Release

HRR (A) and THR (B) curves of bamboo specimens before and after self-assembly with PA-PEI are presented in **Figure 5**. As illustrated in **Figure 5A**, the combustion process of bamboo specimens is mainly divided into two stages. For the first stage, two HRR peaks of the untreated and treated bamboo specimens were observed. Peak HRR (PHRR) of A5 and B1 was greater than that of the control. And the time to PHRR of A5 and B1 was also slightly decreased, indicating the earlier combustion of A5 and B1. This may be due to the dehydration of wood components catalyzed by PA in a low-temperature stage, and the degradation of organic compounds was accelerated, which promoted the release of combustible volatile products (Wang et al., 2004; Gong et al., 2020). Moreover, PHRR of B5 (213.41 kW/m²) was decreased by 18.71% as compared with the control group (262.54 kW/m²). Therefore, it is evident that increasing the concentration of PA-PEI solutions and times of LbL self-assembly with PA-PEI contributes to a slight improvement in the flame retardancy of bamboo specimens in the first combustion stage of cone calorimeter analysis. This may be due to the PA-PEI thin film generated on the surface of treated bamboo specimens in the LbL assembly process with PA-PEI solutions. The PA-PEI thin film can effectively prevent bamboo specimens from combusting with the increase of concentration of PA-PEI solution and times of LbL assembly.

The PHRR in the second stage, average HRR (AHRR), PHRR, and THR of bamboo specimens are shown in **Table 3**. A reduction in PHRR of bamboo specimens after LbL self-

TABLE 3 | AHRR and PHRR of bamboo specimens before and after LbL self-assembly with PA-PEI.

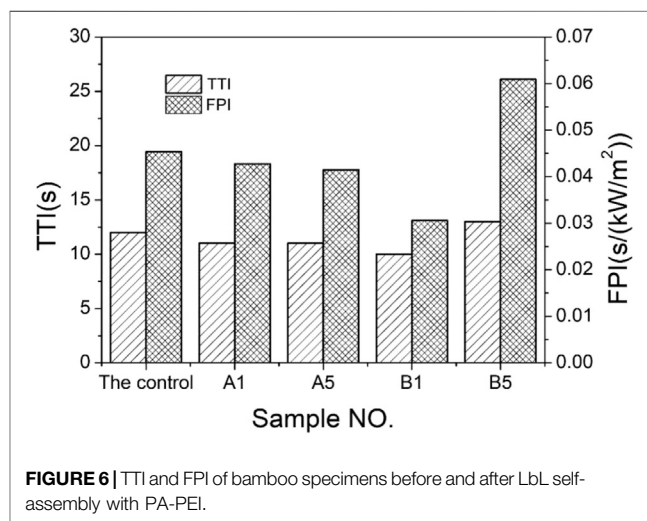
Sample no.	AHRR (kW/m ²)	PHRR ₂ (kW/m ²)	Time to PHRR ₂ (s)	PHRR (kW/m ²)	THR (MJ/m ²)
The control	76.34	264.63	115	264.63	41.67
A1	83.30	257.55	125	257.55	39.24
A5	64.34	245.00	144	265.15	38.02
B1	62.40	276.12	118	327.25	37.21
B5	53.68	212.84	181	213.41	32.38

assembly with PA-PEI was generated as compared to the control. The corresponding values of PHRR₂ were decreased by 2.68% (A1), 11.17% (A5), and 19.57% (B5) in comparison with the control group, respectively. The time to PHRR was delayed from 115 s (the control group) to 125 s (A1), 144 s (A5), 118 s (B1), and 181 s (B5), suggesting lower flammability of the treated bamboo specimens. The reason was that the increasing concentrations of PA-PEI solution and times of LbL self-assembly with PA-PEI resulted in the generation of IFR film on the surface of bamboo specimens, which can protect the underlying bamboo matrix from sharp combustion during the cone calorimeter test. However, PHRR of B5 (213.41 kW/m²) prepared by using the IFR system with 10 wt% PA solution and 10 wt% PEI solution was greater than that of FR wood (164.80 kW/m²) prepared by using 8 wt% PA solution and 3 wt% uracil solution (Zhang L. et al., 2020).

THR curves of bamboo specimens before and after LbL self-assembly with PA-PEI are presented in **Figure 5B**. It was noted that the THR of the treated bamboo specimens was decreased with the increase in the concentration of PA-PEI solution and times of LbL self-assembly. For the bamboo specimens LbL self-assembly with PA-PEI, the following order of THR was found: A1>A5>B1>B5 (**Table 3**). A small decrement of 8.76% (from 41.67 MJ/m² to 38.02 MJ/m²) occurred in the THR of bamboo specimens (A5) self-assembly with the IFR system with 5 wt% PA solution and 5 wt% PEI solution with increase times of LbL self-assembly to five in comparison with the control. Moreover, THR of bamboo specimens (B5) self-assembly with the IFR system with 10 wt% PA solution and 10 wt% PEI solution five times the LbL self-assembly reduced by 22.30% (from 41.67 MJ/m² to 32.38 MJ/m²). It indicated that both improvement in PA-PEI solution and times of LbL self-assembly can help to improve the flame retardancy of bamboo specimens. It was also noted that THR of B5 (32.38 MJ/m²) prepared by using the IFR system with 10 wt% PA solution and 10 wt% PEI solution was greater than that of FR wood (16.90 MJ/m²) prepared by using 8 wt% PA solution and 3 wt% uracil solution (Zhang Y. et al., 2020).

Time to Ignition and Fire Performance Index

Time to ignition (TTI) is the continuous ignition time required from the surface of the tested materials to be ignited under the preset heat radiation power. Fire performance index (FPI) is the ratio of the TTI of the tested material to the PHRR. FPI is calculated according to the following formula: $FPI (s/(kW \cdot m^{-2})) = TTI/PHRR$, which is usually used to evaluate the potential flashover hazard of the material in a fire (Petrella, 1994; Xu et al., 2013; Yu

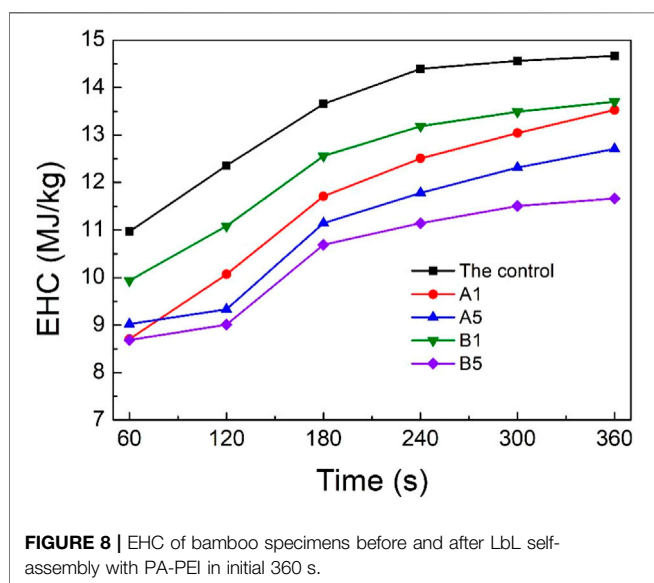
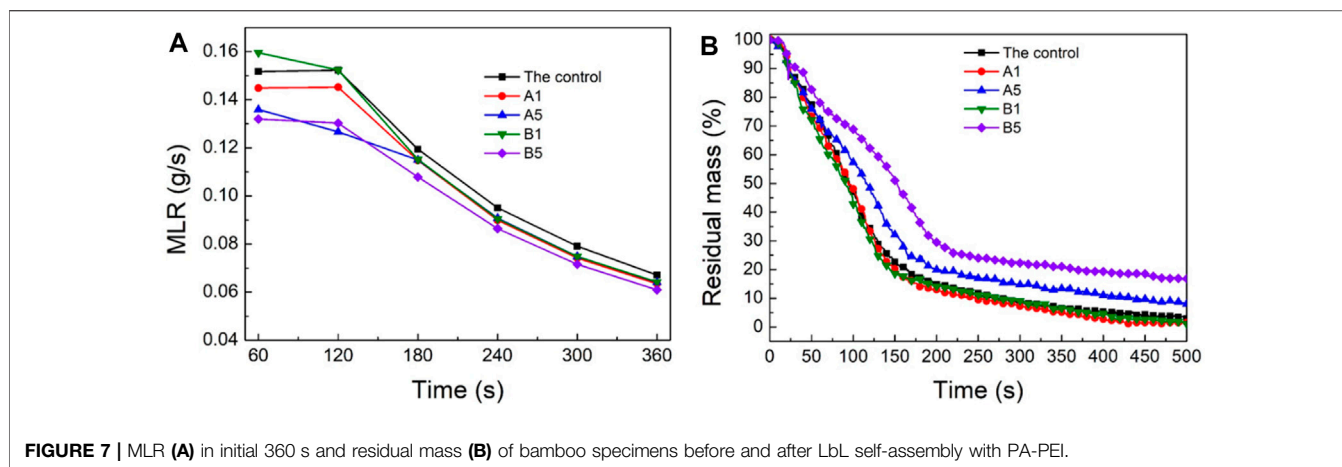
**FIGURE 6** | TTI and FPI of bamboo specimens before and after LbL self-assembly with PA-PEI.

et al., 2016; Hou et al., 2017). Besides, the flashover time of the tested material is an important basis for the design of fire escape time. The larger the FPI, the later the material flashover time.

Figure 6 plots TTI and FPI of bamboo specimens before and after LbL self-assembly with PA-PEI. It indicated that the concentration of PA-PEI solution and times of LbL self-assembly had a limited effect on TTI of bamboo specimens. Additionally, TTI of bamboo specimens was slightly increased from 12 to 13 s with the increasing times of LbL self-assembly to five, with the IFR system with 10 wt% PA solution and 10 wt% PEI solution. However, FPI of B5 was increased by 35.56% (from 0.045 s/(kW/m²) to 0.061 s/(kW/m²)) in comparison to the control. Actually, both increase in TTI and decrease in PHRR contribute to the increase of FPI, further leading to improved fire resistance of bamboo specimens LbL self-assembly with PA-PEI (Hou et al., 2019).

Mass Loss Rate and Residual Mass

MLR in initial 360 s and residual mass of bamboo specimens before and after LbL self-assembly with PA-PEI are presented in **Figure 7**. As shown in **Figure 7A**, MLR of the treated bamboo specimens was decreased with the increase in PA-PEI solution and times for LbL self-assembly with PA-PEI. For the bamboo specimens LbL self-assembly with PA-PEI, the following order of MLR in initial 180 s was found: B1>A1>A5>B5. That of MLR of A1, A5, and B1 was almost the same from 180 to 360 s as presented in **Figure 7A**. Residual mass of A1 (1.54%) and B1 (1.07%) decreased more considerably than that of the control



group (2.90%) as illustrated in **Figure 7B**. Besides, residual mass of A5 (8.07%) was 178.28% greater and B5 (16.84%) was 480.69% greater than the control group (2.90%), respectively. It indicated that the IFR system with PA-PEI solution can exert a flame retardant action at the flaming combustion stage to fix the carbon source, thereby producing more carbon residue (i.e., Residual mass). Additionally, the PA-PEI thin film formed on the surface of the treated bamboo specimens in LbL assembly process can effectively protect bamboo specimens from combusting with increasing times of LbL self-assembly. Similar results were obtained from TG and DTG analysis, as shown in **Figure 4**.

Effective Heat of Combustion, CO Yield, and CO₂ Yield

Figure 8 illustrates the EHC of bamboo specimens before and after LbL self-assembly with PA-PEI in initial 360 s, and the detailed data for the EHC of the bamboo specimens before and after LbL self-assembly with PA-PEI are listed in **Table 4**. As shown in **Figure 8**, the EHC of the treated bamboo specimens was decreased with increasing PA-PEI concentrations and

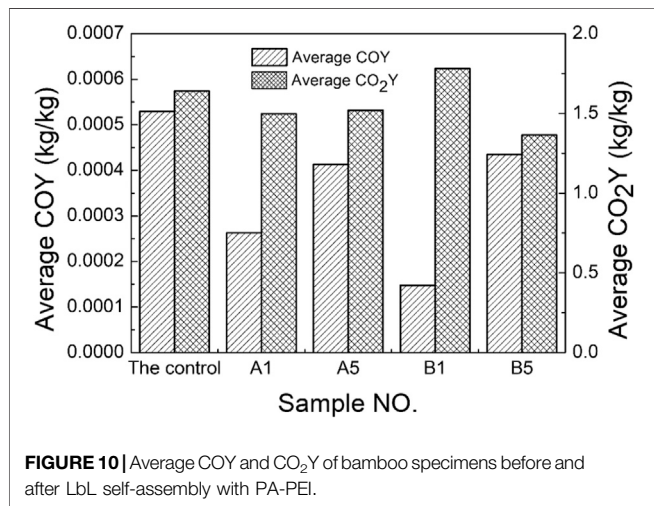
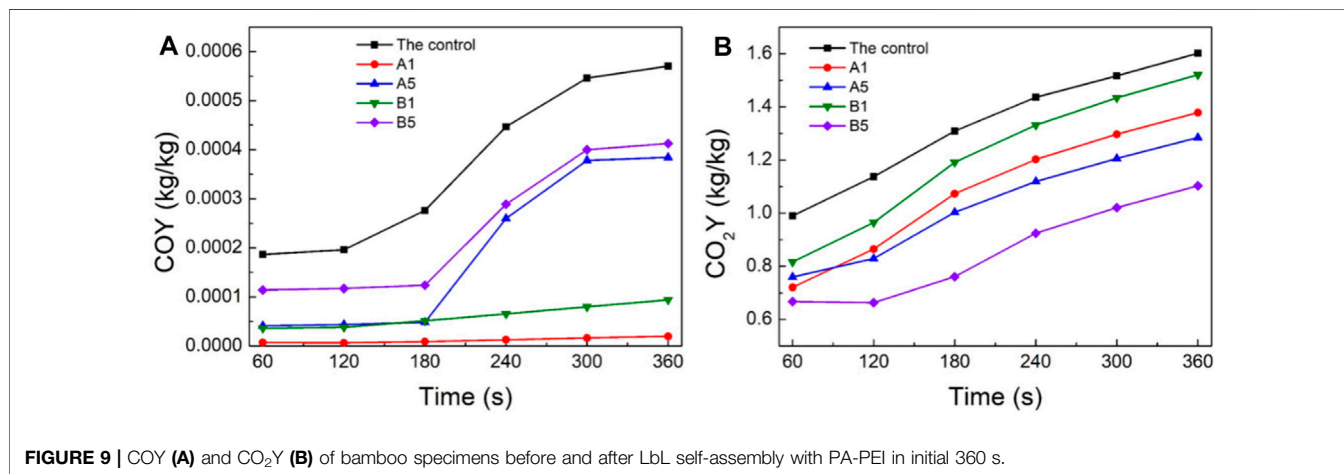
times of LbL self-assembly. Average EHC and peak EHC of bamboo specimens after LbL self-assembly with PA-PEI (i.e., A1, A5, and B1) were slightly decreased by less than 10% as shown in **Table 4**. Besides, average EHC and peak EHC of bamboo specimens after LbL self-assembly with PA-PEI were decreased by 18.90% (B5: 11.84 MJ/kg) with increasing times of LbL self-assembly to five at the IFR system with 10 wt% PA solution and 10 wt% PEI solution in comparison with the control group (14.60 MJ/kg). In addition, the time to peak EHC of the treated bamboo specimens was remarkably prolonged and more than doubled to compare with the control group (**Table 4**), indicating that the IFR system with 10 wt% PA solution and 10 wt% PEI solution plays a significant role in limiting the production of flammable volatile products.

COY and CO₂Y of bamboo specimens before and after self-assembly with PA-PEI in the initial 360 s are presented in **Figure 9**. It was found that the COY was increased with the increase in the concentration of PA-PEI solution and the times of LbL self-assembly with PA-PEI, and the following order of COY in the initial 360 s was found: B5>A5>B1>A1. The reason was that the thin film of PA-PEI formed on the surface of the treated bamboo specimens was thick enough to prevent bamboo specimens from combusting with the increase of concentration of PA-PEI solution and times of LbL self-assembly (Jiang et al., 2012). Hence, a remarkable increase in the COY of the treated bamboo specimens was obtained and increased with the delay of combustion time. However, the following order of CO₂Y in the initial 360 s was found: B1>A1>B5>A5. The incomplete combustion of the treated bamboo specimens led to a reduction in the CO₂Y during the cone calorimeter test.

Average COY and CO₂Y of bamboo specimens before and after self-assembly with PA-PEI are shown in **Figure 10**. The average COY of the treated bamboo specimens was increased with the increase in the times of LbL self-assembly with PA-PEI, and the following order of average COY was found: B5>A5>A1>B1. The average COY of the treated bamboo specimens was decreased by 50.28% (A1), 72.02% (B1), 21.93% (A5), and 17.77% (B5) as compared with the control,

TABLE 4 | EHC of bamboo specimens before and after LbL self-assembly with PA-PEI in initial 360 s.

Sample no.	Average EHC/(MJ/kg)	Peak EHC/(MJ/kg)	Time to peak EHC/s
The control	14.60	74.41	115
A1	14.45	79.78	324
A5	13.54	69.46	551
B1	13.35	78.82	359
B5	11.84	69.94	151



respectively. By contrast, the average CO₂Y of bamboo specimens after self-assembly with PA-PEI (i.e., A1, A5, and B5) was slightly decreased by less than 20%. B5 showed a decrease of 16.88% in average CO₂Y (B5: 1.364 kg/kg) compared to the control group (1.641 kg/kg). It indicated that the IFR system with PA-PEI played an obvious inhibiting effect on the combustion of bamboo materials during the cone calorimeter test (Nguyen et al., 2013). It is well known that bamboo is mainly composed of cellulose, lignin, and hemicellulose, and it will crack to produce small-molecule combustible substances under combustion conditions. Thereby, CO and CO₂ are expected to be generated after the

combustion of bamboo (Jakab et al., 1995; Basilakis et al., 2001). Both increase in average COY and decrease in CO₂Y of the treated bamboo specimens were caused by the imperfect combustion of bamboo specimens LbL self-assembly with PA-PEI. The thin film of PA-PEI formed on the surface of the treated bamboo specimens was thick enough to prevent bamboo specimens from combusting with the increase of concentration of PA-PEI solution and times of LbL self-assembly (Nguyen et al., 2013).

Combined with the analysis of EHC shown in **Figure 8** and **Table 4**, an obvious fixation effect of the IFR system with PA-PEI solution on the C element was obtained in the combustion process of bamboo materials. Additionally, the PA-PEI thin film formed on the surface of treated bamboo specimens in the LbL assembly process was thick enough to protect bamboo specimens from thermal decomposition and sharp combustion, leading to the increase in amount of residual mass (**Figure 7B**) and decrease in EHC (**Figure 8**) during the cone calorimeter test. Similar results were obtained from the TG-DTG analysis of bamboo specimens after self-assembly with PA-PEI. The formation of a protective char layer during the cone calorimeter measurement acts as a barrier, reduces the HRR and the yield of CO₂, and further increases residual mass, indicating that a well-integrated flame retardant performance was obtained to the bamboo specimens treated with the IFR solution of 10 wt% PA and 10 wt% PEI five times the LbL self-assembly with PA-PEI.

CONCLUSIONS

The PA-PEI-based fire-retardant layer was coated on the surface of shaving super bamboo specimens by an LbL self-assembly

technology to improve the flame retardancy of bamboo materials in this study. The effects of PA-PEI solution and times of LbL self-assembly with PA-PEI on the flame retardancy of bamboo specimens were systematically investigated. The LbL self-assembly with PA-PEI promoted the generation of a residual char layer with excellent stability. In addition, a higher solution concentration of PA-PEI and more times of LbL self-assembly made the specimens exhibit smaller HRR and lower yield of CO and CO₂, but greater residual mass and FPI values. For bamboo specimens self-assembly with 10 wt% PA solution and 10 wt% PEI solution five times, PHRR and THR were reduced by 19.36 and 22.30%, respectively, compared to the control group. In addition, the FPI and residual mass of the bamboo specimens self-assembly with 10 wt% PA solution and 10 wt% PEI solution 5 times were increased by 35.56 and 480.70% in comparison with the control group, respectively. This work contributes to a better understanding of the effects of LbL self-assembly with PA-PEI on the flame retardancy of bamboo specimens, thus providing a useful approach for the value-added utilization of bamboo materials.

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DATA AVAILABILITY STATEMENT

The original contribution presented in the study are included in the article/supplementary material; further inquiries can be directed to the corresponding authors.

AUTHOR CONTRIBUTIONS

PL, YJ, and JH executed the experiment and wrote the manuscript. YY conceived and designed the study and experiment plan. LM and WC revised the manuscript. XR and XZ analyzed the experimental results.

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Conflict of Interest: PL was employed by the company Zhonghang Monitoring and Testing Technology Research Institute Co., Ltd.

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