

Flame-Retardancy and Smoke Suppression Characteristics of Bamboo Impregnated with Silicate-Intercalated Calcium Aluminum Hydrotalcates

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Flame-retardant silicate-intercalated calcium aluminum hydrotalcites (CaAl-SiO₃-LDHs) were synthesized to treat bamboo for retardancy, to overcome the bamboo's flammability and reduce the production of toxic smoke during combustion. The microstructure, elemental composition, flame retardancy, and smoke suppression characteristics of the bamboo before and after the fire-retardant treatment with different pressure impregnation were studied using a scanning electron microscope (SEM), elemental analysis (EDX), and cone calorimetry. It was found that CaAl-SiO₃-LDHs flame retardants can effectively fill and cover the cell wall surface and the cell cavity of bamboo without damaging the microstructure. As compared to the non-flame-retardant bamboo, the heat release rate (HRR) of the CaAl-SiO₃-LDHs flame-retardant bamboo was significantly reduced, the total heat release (THR) decreased by 31.3%, the residue mass increased by 51.4%, the time to ignition (TTI) delay rate reached 77.8%, the mass loss rate (MLR) decreased, and the carbon formation improved. Additionally, as compared to the non-flame-retardant bamboo, the total smoke release (TSR) of the CaAl-SiO₃-LDHs flame-retardant bamboo decreased by 38.9%, and the carbon monoxide yield (Y_{CO}) approached zero. Thus, the CaAl-SiO₃-LDHs flame-retardant bamboo has excellent flame-retardancy and smoke suppression characteristics.

Keywords: Layered double hydroxides (LDHs); Silicate intercalation; Calcium aluminum hydrotalcite; Bamboo; Flame-retardant; Smoke suppression; Characteristics

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INTRODUCTION

Bamboo and products that use bamboo as a raw material, such as thin sliced bamboo, bamboo flooring, and bamboo laminated timber, have been widely used in the fields of construction, decoration, and furniture manufacturing. However, bamboo and its products are easy to burn and conducive to building fires, thus likely to cause a large number of casualties and huge property losses (Du *et al.* 2017a,b). Bamboo can be converted into a flame-retardant material if flame-retardant treatment is applied, which is of great significance in reducing the occurrence of fire and enhancing safety in the use of the bamboo. In recent years, the research and application of the bamboo flame-retardants have received much attention, and the number of research reports on the bamboo flame-retardants has been increasing (Li *et al.* 2017; Zhang *et al.* 2018; Li *et al.* 2019; Lin *et al.* 2019). Flame retardant bamboo is usually prepared by applying flame retardant to bamboo (Zhou *et al.* 2016); however, most of the flame-retardants used in the bamboo now, which

contain phosphorus, nitrogen, boron, and other elements, are water-based inorganic materials (Jin *et al.* 2015; Zheng *et al.* 2016; Jin *et al.* 2019). These flame retardants have shortcomings such as strong moisture absorption, which causes the retardant to precipitate from the surface of the bamboo (Zhou *et al.* 2018), reducing the flame-retardant properties of bamboo. Therefore, it is not only necessary but also important to develop new bamboo flame-retardants that are not likely to be lost or precipitated from the surface of the bamboo.

The emergence of hydrotalcite flame-retardants has made it possible to overcome such disadvantages by providing new solutions. Hydrotalcite and hydrotalcite-like compounds are also called layered bimetallic hydroxides (LDHs), which are characterized by controllable interlayer anions and rich hydroxyl groups on the surface of the laminates (Zhou *et al.* 2013; Yao *et al.* 2019a). As a flame-retardant, LDHs are characterized by their non-toxicity, absence of halogen, and excellent flame-retardant and smoke suppression performance (Guo *et al.* 2017; Xu *et al.* 2018). Therefore, the bamboo applied with hydrotalcite flame-retardant treatment not only shows better flame-retardant and smoke-suppressing performance, but it also forms a hydrogen bond with the hydroxyl group of hydrotalcite. As a result, the flame-retardants are fixed into the bamboo and are not easily lost and precipitated. However, at present, there have been very few research reports on hydrotalcite flame-retardant bamboo, and the preliminary study on MgAl-LDHs for the flame-retardant bamboo has been conducted only by an author of this paper (Yao *et al.* 2019a,b). However, in his previous preliminary study, the author found that the preparation cost of the flame-retardant MgAl-LDHs was relatively high (Hua *et al.* 2019), which is not considered conducive to its popularization and application. Therefore, calcium, which is cheap and abundant, was selected as the divalent cation of the hydrotalcite laminate, and the silicate as the intercalated anion. In this way, silicon was introduced into hydrotalcite, and a relatively cheap silicate-intercalated calcium aluminum hydrotalcite (CaAl-SiO₃-LDHs) was synthesized. CaAl-SiO₃-LDHs were then used to process the bamboo for flame retardancy. The flame-retardant and smoke-suppressing properties of bamboo before and after applying the flame-retardant treatment were studied, which provides a new method for the development of bamboo flame-retardants.

EXPERIMENTAL

Materials

Ca(NO₃)₂·6H₂O, Al(NO₃)₃·9H₂O, Na₂SiO₃·9H₂O, and NaOH were analytically pure (AR) and purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China. Additionally, the deionized water was self-prepared.

Bamboo strips were made from moso bamboo, with a size of 100 mm × 20 mm × 5 mm (length × width × thickness) parallel to the longitudinal plane of the grain direction, without bamboo knots, and with a moisture content of about 10%. The moso bamboo was purchased from Zhejiang Yongyu Bamboo Joint-Stock Co., Ltd., Huzhou, China.

Synthesis of the CaAl-SiO₃-LDHs

The drug metering was determined based on the general formula of hydrotalcite ($[M_{1-x}^{2+}M_x^{3+}(OH)_2](A_{x/n}^{n-}) \cdot mH_2O$). The amounts of Ca(NO₃)₂·6H₂O and Al(NO₃)₃·9H₂O were weighed according to the molar ratio of Ca²⁺:Al³⁺ = 2. The molar concentration of Ca²⁺ was 0.33 mol/L. An appropriate amount of deionized water was added to dissolve the

aforementioned substances in a beaker for preparing a mixed salt solution. The corresponding amount of $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ was weighed and then dissolved with an appropriate amount of deionized water. High-concentration NaOH solution was prepared for standby. The sodium silicate solution was transferred to a 500 mL three-port flask, and the flask was placed at 25 °C in a constant-temperature oil bath pot. The device was turned on and began stirring. The prepared mixtures of the salt solution and the NaOH solution were transferred to a constant-pressure dropping funnel, respectively. First, the NaOH solution was dropped into the flask until the pH of the solution reached 10. Then, the mixed salt solution was dropped into the flask, and the potential of hydrogen (pH) of the solution was maintained at 10, after which the white slurry was prepared. Thereafter, a crystallization reaction was carried out on the white slurry at a reaction temperature of 100 °C with constant and intense stirring. After 9 h of crystallization, the reaction system was put under static aging at room temperature for 12 h, and N_2 was used for protection throughout the crystallization process. Finally, the CaAl-SiO₃-LDHs powder was obtained by filtration, washing, drying, and grinding.

CaAl-SiO₃-LDHs Flame-Retardant Treated Bamboo

The prepared CaAl-SiO₃-LDHs powder was dispersed in an aqueous solution to prepare a suspension at 1 wt.% by ultrasonic (1 h at room temperature). The bamboo strips were then placed and completely immersed in the CaAl-SiO₃-LDHs suspension. Then, the suspension was placed in a pressurized tank and was sealed, and thereafter it was vacuumed. After the vacuum degree reached 0.085 MPa, the tank pressure was maintained for 20 min. Then, different pressures of 0.2 MPa, 0.4 MPa, and 0.6 MPa, respectively, were applied, and the bamboo strips were impregnated. When the pressure reached the preset value, the exhaust valve was opened to relieve the pressure, and then the system was again pressurized to the preset value. This treatment was repeated several times in order to impregnate the bamboo strips for 3 h. Finally, the bamboo strips were taken out. After the surface suspension was washed with deionized water, and the strips were dried in a drying oven, the CaAl-SiO₃-LDHs flame-retardant treated bamboo was prepared.

Analysis and Testing

Cold field emission scanning electron microscopy (SEM) analysis

Flame-retardant bamboo and non-flame-retardant bamboo was sliced. A SU8010 cold field emission scanning electron microscope (SEM; Hitachi, Tokyo, Japan) was applied, and thereafter the microstructure of the bamboo before and after applying the flame-retardant treatment was analyzed at an accelerating voltage of 25.0 kV.

Energy-dispersive X-ray (EDX) analysis

The flame-retardant treated bamboo was sliced, and its elemental composition was observed and analyzed at an accelerating voltage of 25 kV using the SIRION-100 scanning electron microscope (Ametek, Newark, DE, USA) and Genesis 4000 energy-dispersive X-ray instrument (Ametek, Newark, DE, USA).

Cone calorimeter test

The non-flame-retardant bamboo and the flame-retardant treated bamboo were respectively bonded into test pieces with a size of 100 mm × 100 mm × 5 mm (length × width × thickness). In order to reduce of dissipation of the burning heat and affect the experiment, the five sides of each sample except for the heating surface were covered with

tin foil, and an asbestos pad was placed under the sample to prevent heat loss from the bottom of the sample, with the uncoated surface of the test piece facing upward. The test piece and the asbestos pad were placed together in a stainless steel mold box, and the surface of the test piece was covered with a wire mesh and fixed by mold lid. Then, according to the ISO5660-1 procedure, test pieces were tested for combustion performance with a cone calorimeter (Fire Testing Technology Ltd., East Grinstead, UK). The heat radiation power was 50 kWm^{-2} (Li *et al.* 2002), and the corresponding temperature was 728°C . Finally, the experimental data were collected, processed, and analyzed using the software dedicated to cone calorimeter analysis.

RESULTS AND DISCUSSION

SEM Analysis

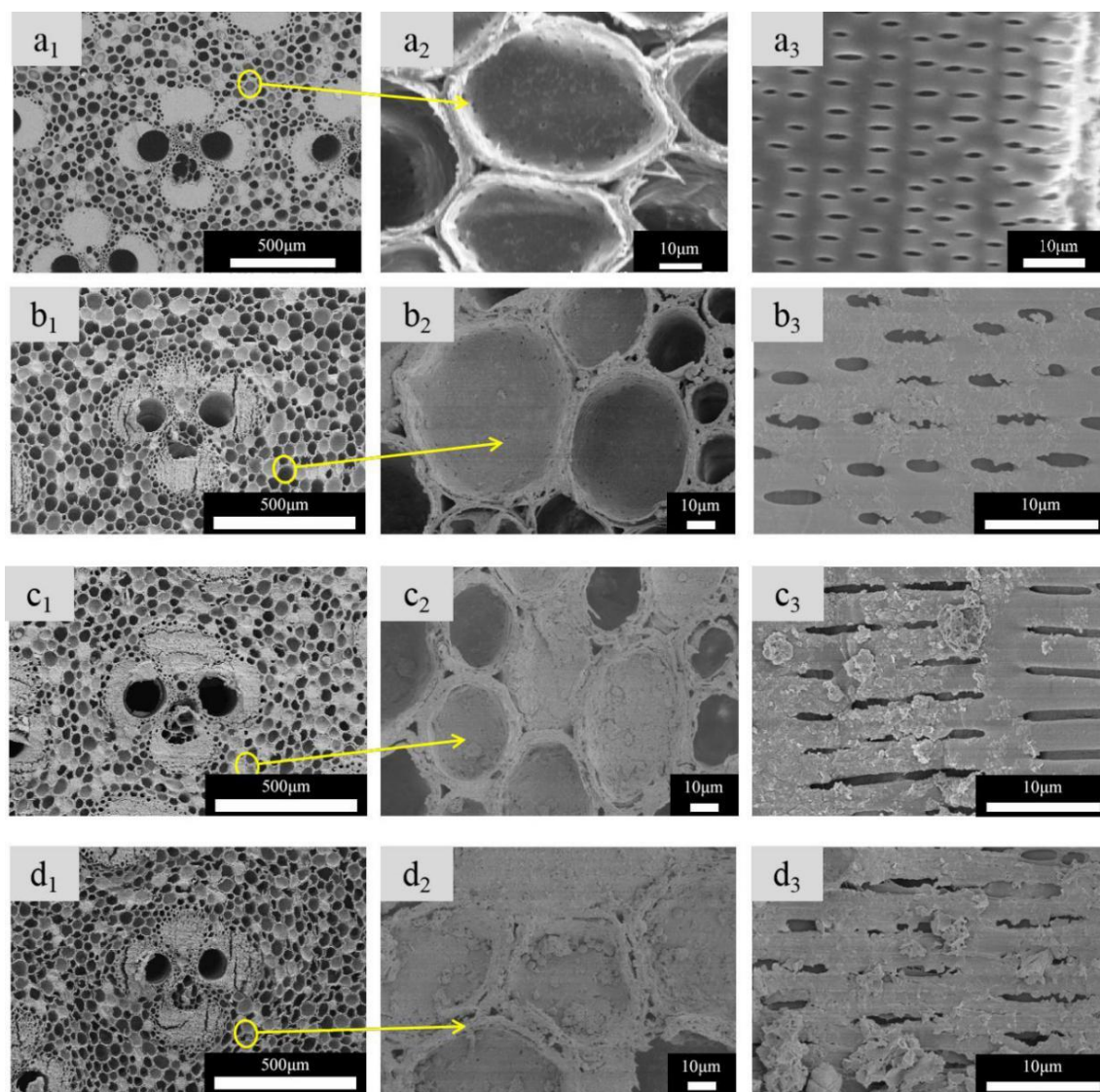


Fig. 1. SEM diagram of the non-flame-retardant bamboo (a₁₋₃) and the flame-retardant bamboo (b₁₋₃) applied with a pressure of 0.2 MPa, the flame-retardant bamboo (c₁₋₃) applied with a pressure of 0.4 MPa, and the flame-retardant bamboo applied with a pressure of 0.6 MPa (d₁₋₃)

The cold field emission scanning electron microscope (SEM) was used to observe the microstructure of the bamboo before and after applying the CaAl-SiO₃-LDHs flame-retardant treatment, and the corresponding results are shown in Fig. 1. Among them, Figs. a₁₋₂, b₁₋₂, c₁₋₂, and d₁₋₂ show the cross-section of the bamboo, whereas Figs. a₃, b₃, c₃, and d₃ show the radial section of the bamboo.

Comparing the SEM diagram of the cross-sections of the non-flame-retardant bamboo and the flame-retardant bamboo (Figs. a₁, b₁, c₁, and d₁), it can be seen that the vascular bundles and parenchyma cells, *etc.*, of the bamboo after the CaAl-SiO₃-LDHs flame-retardant treatment still maintained the original microstructure and form and had not been destroyed. From the partially enlarged SEM diagram of the cross-section of the flame-retardant bamboo (Figs. b₂, c₂, and d₂), it is easy to observe that the surface of the cell wall and the cell cavity of the flame-retardant bamboo were filled or covered with a large amount of CaAl-SiO₃-LDHs flame-retardants, and the filled or the covered area showed an increase as shown in Figs. b₂, c₂, and d₂. Also, from the radial cross-section diagram of the bamboo before and after applying the flame-retardant treatment (Figs. a₃, b₃, c₃, and d₃), it can be seen that the cell wall surface and pores of the flame-retardant bamboo were filled or covered with a large amount of CaAl-SiO₃-LDHs flame-retardants with an increased filled/covered area, as shown in Figs. b₃, c₃, and d₃. It was discovered that the CaAl-SiO₃-LDHs flame-retardants could effectively enter the bamboo cell cavity and adhere to the bamboo cell wall after the bamboo was treated with pressure impregnation, and an increased drug loading capacity of the flame-retardant was observed with an increase in pressure. Thus, this kind of treatment is considered an excellent method for the preparation of the CaAl-SiO₃-LDHs flame-retardant bamboo.

EDX Analysis

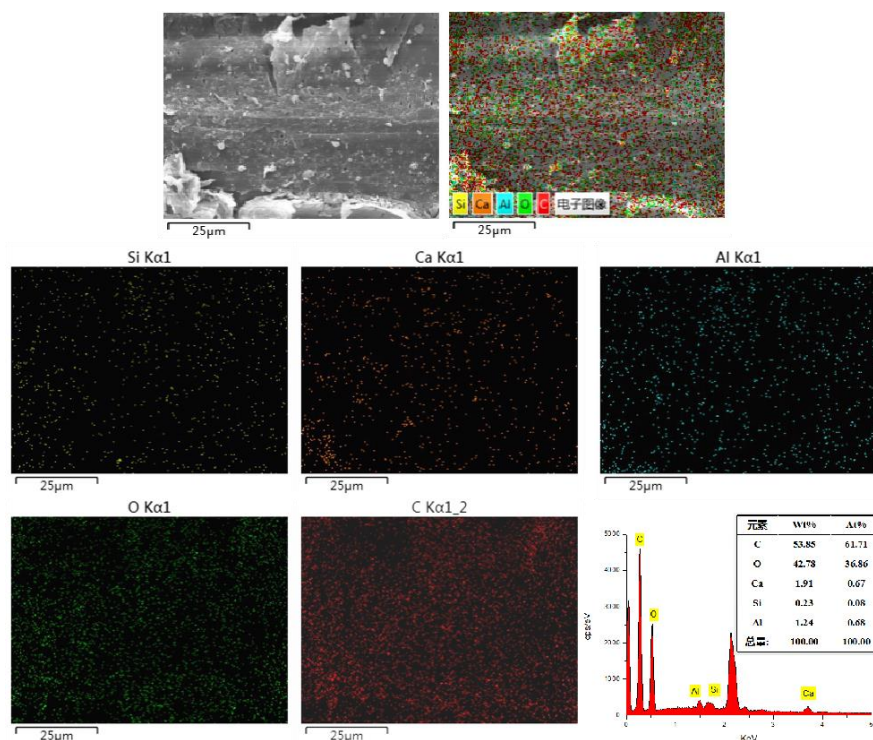


Fig. 2. EDX map and spectrum of the flame-retardant bamboo

The energy-dispersive X-ray (EDX) was used to observe and analyze the elementary composition of the bamboo impregnated with the CaAl-SiO₃-LDHs flame-retardant under a pressure of 0.6 MPa. The results are shown in Fig. 2. After the CaAl-SiO₃-LDHs flame-retardant treatment, the characteristic peaks of both the main elements of the bamboo (C and O) and those of CaAl-SiO₃-LDHs (Ca, Al, and Si) were apparent. Therefore, CaAl-SiO₃-LDHs can be effectively loaded on the bamboo with the flame-retardant treatment of pressure impregnation, which is found to be consistent with the SEM result and shows that the cell cavity and the cell wall of the bamboo were filled or covered with a large amount of CaAl-SiO₃-LDHs flame-retardants.

Heat Release Rate (HRR)

With CaAl-SiO₃-LDHs as the flame retardant, the bamboo was subjected to pressure impregnation treatment with different pressures. The heat release rate (HRR) curves of the non-flame-retardant bamboo and the flame-retardant bamboo are shown in Fig. 3.

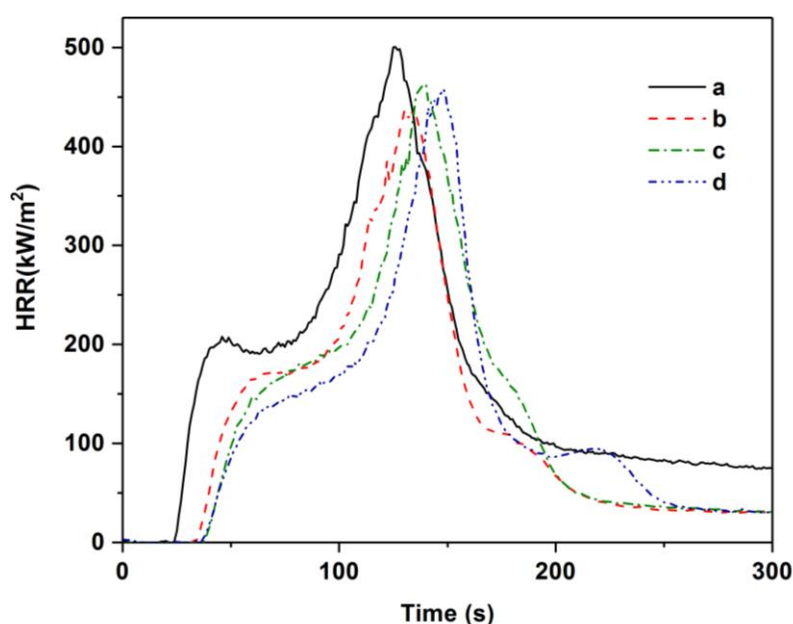


Fig. 3. HRR of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2MPa (b), 0.4MPa (c), and 0.6MPa (d)

HRR is also called fire intensity. Higher HRR indicates a greater risk of fire. Conversely, the smaller the HRR, the less likely the material is to catch fire, and thus the better the flame-retardant performance. Therefore, a reduction in the HRR can effectively reduce the fire hazard of a material. Generally, the two exothermic peaks of the HRR curve correspond to the short flame combustion at the point of material ignition and the combustion, respectively, when a higher flame occurs (Du *et al.* 2017b). Therefore, bringing down the peak value of the second exothermic peak is considered an effective method of improving the flame-retardant performance of the material. As can be seen from Fig. 3, as compared to the non-flame-retardant bamboo, the values of the two exothermic peaks of the 0.2 MPa pressurized flame-retardant bamboo were decreased by 17.9% and 12.7%, respectively. The values of the two exothermic peaks of the 0.4 MPa pressurized flame-retardant bamboo were decreased by 22.8% and 8.1%, respectively, and the values

of the two exothermic peaks of the 0.6 MPa pressurized flame-retardant bamboo were decreased by 31.6% and 8.4%, respectively. It can be seen that both the first exothermic peak and the second exothermic peak of the flame-retardant bamboo showed values smaller than those of the non-flame-retardant bamboo. Therefore, the flame-retardant performance of the bamboo after the CaAl-SiO₃-LDHs treatment was evidently improved. This was primarily due to the fact that addition of the CaAl-SiO₃-LDHs flame-retardant inhibited the speed of the heat release and decreased the combustion rate of the material.

Total Heat Release (THR)

After pressure impregnation, the total heat release (THR) curve of non-flame-retardant bamboo and flame-retardant bamboo tested by the cone calorimeter is shown in Fig. 4.

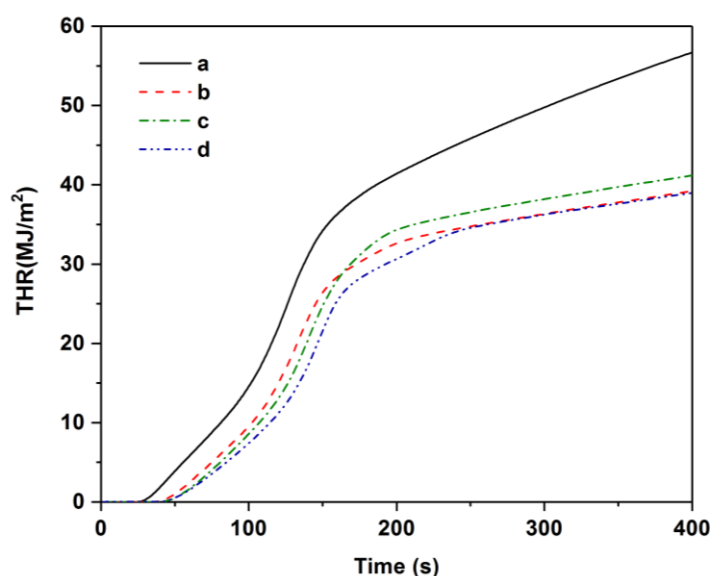


Fig. 4. THR of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2MPa (b), 0.4MPa (c), and 0.6MPa (d)

The THR indicates the total amount of heat released by the material during the combustion process. A greater THR means a greater risk of the material to catch fire (Chen *et al.* 2019). Therefore, a reduction in the total amount helps in delaying material combustion. From Fig. 4, it can be seen that throughout the combustion process the THR of the bamboo material treated with the pressure impregnation of CaAl-SiO₃-LDHs was less than that of the non-flame-retardant material. As compared to the non-flame-retardant bamboo, the final THR value of the 0.2 MPa pressurized flame-retardant bamboo decreased by 30.9%, the 0.4 MPa pressurized flame-retardant bamboo decreased by 27.5%, and the 0.6 MPa pressurized flame-retardant bamboo decreased by 31.3%. Therefore, the flame-retardant performance of the bamboo was distinctly improved after applying the flame-retardant treatment with CaAl-SiO₃-LDHs.

Time to Ignition (TTI)

After pressure impregnation, the time to ignition (TTI) curve of non-flame-retardant bamboo and flame-retardant bamboo tested by the cone calorimeter is shown in Table 1.

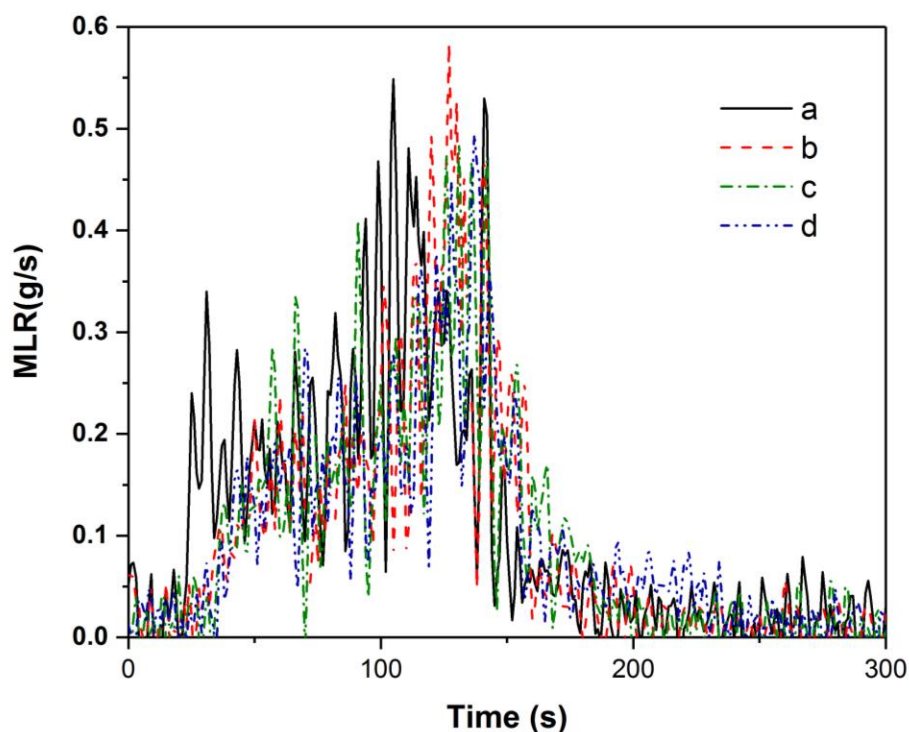
Table 1. TTI of the Non-flame-retardant Bamboo and the Flame-Retardant Bamboo Pressurized under Different Impregnation

Name	Non-Flame-Retardant Bamboo	Pressure of Impregnation (MPa)		
		0.2	0.4	0.6
TTI(s)	18	28	32	32
Delay Rate of TTI(s)	-	55.56%	77.78%	77.78%

The time to ignition (TTI) refers to the time of continuous ignition required by the flame combustion on the material surface (Huang and Guan 2017). The longer the TTI is under the experimental conditions, the more difficult to ignite the material, and thus the better the flame-retardant performance of the material (Bakhtiyari *et al.* 2015). As can be seen from Table 1, as compared to the non-flame-retardant bamboo, the TTI of the flame-retardant bamboo improved significantly, and the ignition process was delayed. With an increase in the impregnation pressure, the TTI of the flame-retardant bamboo was also increased. The TTI of the 0.6 MPa pressurized flame-retardant bamboo was about 1.78 times that of the non-flame-retardant bamboo with a TTI delay rate of 77.8%. Therefore, after the fire-retardant impregnation treatment of CaAl-SiO₃-LDHs, the flame-retardant performance of the bamboo was obviously improved, and thus it was unlikely to ignite.

Mass Loss Rate (MLR)

The mass-loss rate (MLR) curves of the non-flame-retardant bamboo and the flame-retardant bamboo are shown in Fig. 5.

**Fig. 5.** Mass loss rate of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2 MPa (b), 0.4 MPa (c), and 0.6 MPa (d)

The mass loss rate is the rate of change of the mass loss of the material during combustion, which reflects the thermal cracking rate and behavior of the materials under a certain fire intensity. The MLR of the flame-retardant bamboo was lower than that of the non-flame-retardant bamboo, which indicates that the flame-retardant performance of the bamboo treated with the CaAl-SiO₃-LDHs flame-retardant was improved. Moreover, when the MLR curve was compared with the HRR curve, it was observed that the two curves showed a similar change pattern, which indicates that the HRR and MLR of the non-flame-retardant bamboo and the flame-retardant bamboo were basically in a synchronized pace during combustion. In addition, a significant fluctuation was observed in the MLR curve, as shown in Fig. 5. This may be due to the innate anisotropy, the uneven density distribution, of the bamboo. The characteristic of the uneven density distribution was then verified with the non-flame-retardant bamboo and the flame-retardant bamboo, which showed time-varying MLR in the combustion test and thus greater fluctuations in MLR curves.

Residual Mass and Residual Carbon Morphology

The curves with the time-varying residual mass of the non-flame-retardant bamboo, the flame-retardant bamboo, and the carbon residue morphology are shown in Figs. 6 and 7, respectively. As shown in Fig. 6, the residual mass of the flame-retardant bamboo was larger than that of the non-flame-retardant bamboo, and it increased with an increase in the pressure applied in the pressure impregnation treatment of CaAl-SiO₃-LDHs flame-retardants. The residual mass of the non-flame-retardant bamboo was only 8.00% of the initial mass, whereas the residual masses of the bamboo treated with the pressure impregnation of 0.2, 0.4, and 0.6 MPa were 15.76%, 16.13%, and 16.54% of the initial mass, respectively, which shows an increase of 48.9%, 50.1%, and 51.4%, respectively. The amount of charcoal, the primary residue produced after the burning of bamboo, was clearly increased after the bamboo was processed under fire-retardant treatment.

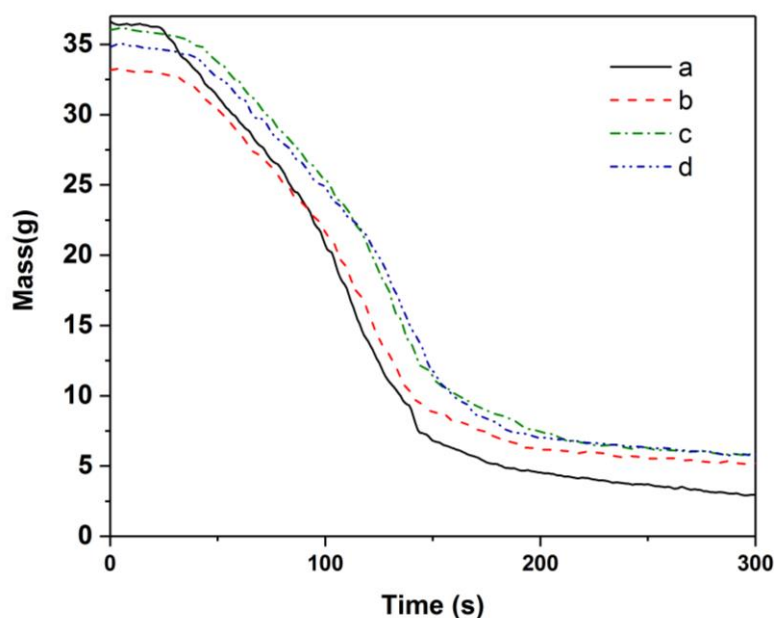


Fig. 6. The residual mass of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2 MPa (b), 0.4 MPa (c), and 0.6 MPa (d)

Figure 7 shows that the carbon residue of the non-flame-retardant bamboo contained a lot of ash, and the shape of the carbon residue was fragmented. The ash content of the carbon residue of the flame-retardant bamboo was less than that of the non-flame-retardant bamboo, and the carbon residue maintained a higher degree of its original shape. Therefore, the carbon residue rate after bamboo burning was improved when the bamboo material was impregnated with the CaAl-SiO₃-LDHs flame-retardant, which shows that CaAl-SiO₃-LDHs induced the effect of promoting char formation (Rasbash 1983). Therefore, the CaAl-SiO₃-LDHs flame-retardant bamboo exhibited an excellent flame-retardant carbon-forming property.



Fig. 7. Diagram of the carbon residue of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2 MPa (b), 0.4 MPa (c), and 0.6 MPa (d)

Total Smoke Release (TSR)

The time-varying curves of the total smoke release (TSR) of the non-flame-retardant bamboo and the flame-retardant bamboo are shown in Fig. 8. The TSR is an important criterion for judging the fire hazard during material combustion. In cases of fires, 75% of the casualties are caused by fire smoke (Stec and Hull 2011), so to reduce the TSR during the combustion of materials provides a practical way to reduce casualties in fires (Du *et al.* 2017a). Fig. 8 shows that the TSR of the flame-retardant bamboo was lower than that of the non-flame-retardant bamboo throughout combustion and demonstrated a decrease in quantity with an increase in the pressure applied in the pressure impregnation treatment of the CaAl-SiO₃-LDHs fire-retardant on the bamboo. As compared to the non-flame-retardant bamboo, the TSR of the bamboo treated with the pressure impregnation of 0.4 MPa and 0.6 MPa was decreased by 15.9% and 38.9%, respectively. Thus, the CaAl-SiO₃-LDHs flame-retardant bamboo exhibited excellent smoke suppression performance.

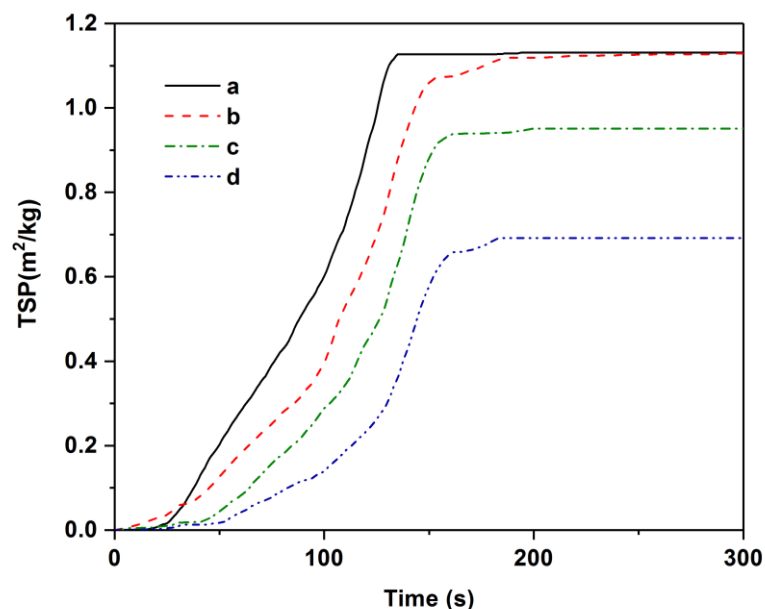


Fig. 8. Total smoke release of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2 MPa (b), 0.4 MPa (c), and 0.6 MPa (d)

Yield of CO

The time-varying curves of the yield of CO (Y_{CO}) of the non-flame-retardant bamboo and the flame-retardant bamboo are shown in Fig. 9.

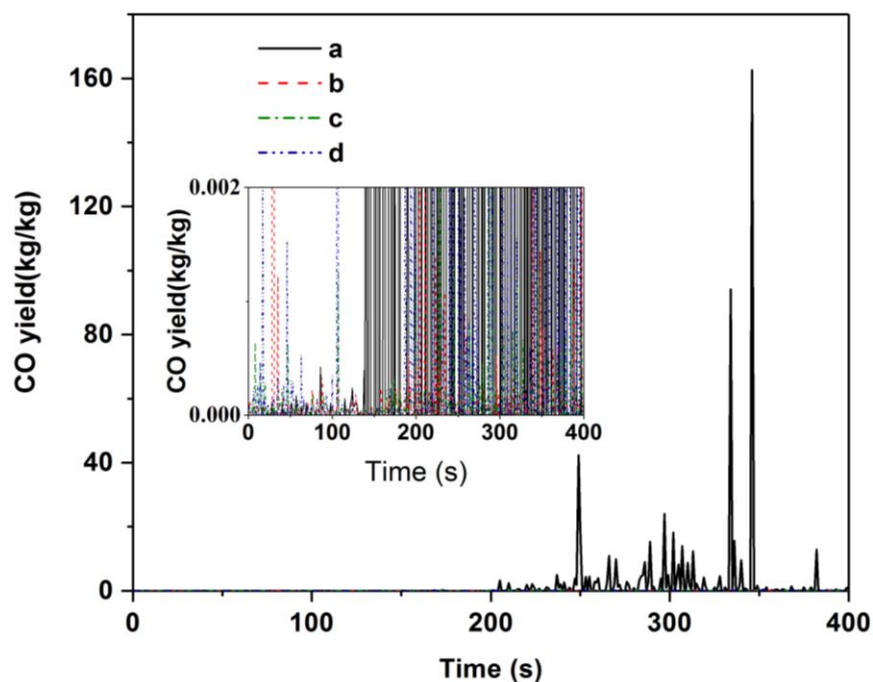


Fig. 9. Carbon monoxide yield of the non-flame-retardant bamboo (a) and the flame-retardant bamboo pressurized of 0.2 MPa (b), 0.4 MPa (c), and 0.6 MPa (d)

The value of Y_{CO} refers to the mass of CO gas produced by the per unit consumption of the material specimen, which is considered an important indicator of the smoke toxicity of materials in combustion. The greater the Y_{CO} , the greater the smoke toxicity (Wang *et al.* 2002). Therefore, it is of great significance to reduce the Y_{CO} of materials in combustion. For the CaAl-SiO₃-LDHs flame-retardant bamboo material, the Y_{CO} of bamboo in the combustion test approached zero in both the flame- and flameless-combustion stages. However, the Y_{CO} of the non-flame-retardant bamboo was close to zero in the flame-combustion stage, but it was found to be higher in the flameless combustion stage. Therefore, the pressure impregnation treatment of the CaAl-SiO₃-LDHs flame-retardant of the bamboo can noticeably reduce the smoke toxicity of bamboo in combustion and thus improves safety while using the bamboo.

CONCLUSIONS

1. The bamboo pieces, after applying the CaAl-SiO₃-LDHs flame-retardant treatment, were maintained in the original microstructure and morphology of their vascular bundles and parenchyma cells, and they suffered no destruction. The flame-retardants could effectively enter the bamboo cell cavity and adhere to the bamboo cell wall, indicating that as the pressure increases, the load of the flame retardant increases.
2. As compared to the non-flame-retardant bamboo, the values of the two exothermic peaks of the 0.6 MPa pressurized flame-retardant bamboo were decreased by 31.6% and 8.4%, respectively, the value of the total heat release (THR) decreased by 31.3%, the time to ignition (TTI) delay rate reached 77.8%, the residue mass increased by 51.4%, and the mass loss rate (MLR) decreased, whereas the carbon formation improved. The findings showed that CaAl-SiO₃-LDHs flame-retardant bamboo exhibited excellent flame-retardant properties.
3. As compared to the non-flame-retardant bamboo, the total smoke release (TSR) of bamboo after applying the 0.6 MPa flame-retardant treatment was decreased by 38.9%, the carbon monoxide yield (Y_{CO}) approached zero, and the smoke toxicity of the flame-retardant bamboo in combustion was significantly reduced. It means that the CaAl-SiO₃-LDHs flame-retardant bamboo has excellent smoke suppression performance.

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