

A Two-step Method for Fabricating Bamboo Culm Coated with MgAl-LDHs and its Fire Resistance Properties

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To enhance the fire-resistance of bamboo, the bamboo was first exposed to sodium hydroxide solution, and then immersed in a suspension of magnesium aluminum layered double hydroxides (MgAl-LDHs) to fabricate modified bamboo (NLB) with a coating of MgAl-LDHs on the surface. X-ray photoelectron spectroscopy showed that the surface of bamboo was coated by the MgAl-LDHs after modification. Fourier transform infrared spectroscopy analysis demonstrated that more hydroxyl groups were available on the bamboo surface after pretreatment with sodium hydroxide solution. The morphology of the MgAl-LDHs coating was firmer and denser with evidence from scanning electron microscopy and energy dispersive X-ray spectrometry. Leaching resistance was thereby enhanced. In addition, the cone calorimeter results showed that the peak heat release rate of the NLB was 29.1% lower than that of untreated bamboo, the total heat release decreased by 24.4%, the smoke release rate and total smoke release decreased by 40.5% and 47.8%, respectively, and the peak of carbon monoxide production decreased by 26.7%. The maximum average heat dissipation rate decreased by 24.0%, and the shape of residual charcoal of the NLB was more complete than that of untreated bamboo after fire. Therefore, NLB has excellent fire resistant and smoke suppression properties.

Keywords: Layered double hydroxides; Two-steps method; Coating; Bamboo; Fire resistance

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INTRODUCTION

Bamboo is a kind of lignocellulosic natural material that poses no problems for the ecological environment. Compared with trees, bamboo has a short growth cycle, high yield, and good mechanical properties. Therefore, bamboo has been widely used in construction and decoration and is becoming increasingly popular (Du *et al.* 2016). However, its use is limited by its combustibility, especially in architecture, leading to greater interest in the study of fire retardancy in bamboo.

Layered double hydroxide (LDH), also known as hydrotalcite-like compounds or anionic clays, consists primarily of hydroxides in the layer and anions between the layers. Currently, LDH has been demonstrated to have good performance in flame retardancy and smoke suppression (Gao *et al.* 2014; Dutta *et al.* 2018; Yao *et al.* 2018). In addition, LDH has the advantage of being non-toxic and halogen-free (Qian *et al.* 2017). Therefore, LDH has attracted widespread attention in the field of flame retardancy. Wang *et al.* (2018a) reported that a ZnAl-LDHs coating that is synthesized on the wood surface under ambient

temperature conditions can enhance the fire resistance of treated wood. Using a two-step method, Guo *et al.* (2017) created MgAl-LDHs coating on the wood surface in a high-pressure reactor at 100 °C. Wang *et al.* (2018b) covered the surface of medium density fiberboard (MDF) with MgAl-LDHs fire retardant. The existing preparation and processing methods of LDHs are complicated and difficult to apply to bamboo. Therefore, a simple and effective method for coating the surface of bamboo with synthesized MgAl-LDHs is needed urgently.

In this study, the MgAl-LDHs was synthesized by hydrolysis action using urea, and then applied to the surface of bamboo *via* a two-step method at normal room temperature and pressure. The dense coating of MgAl-LDHs fire retardant was prepared on the bamboo surface. Compared with the above-mentioned application of LDH as a wood flame retardant, the two-step method has the advantages of simplicity and convenience regarding the preparation of the coating on bamboo surface. In addition, the coated bamboo (NLB) demonstrated an excellent performance of both fire retardancy and smoke suppression. This study could provide a new method for the research of fire retardancy on bamboo.

EXPERIMENTAL

Sample Preparation

Mg(NO₃)₂·6H₂O, (NH₂)₂CO, and NaOH were purchased from the Sinopharm Chemical Reagent Co. Ltd., Shanghai, China. The Al(NO₃)₃·9H₂O was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd., Shanghai, China. Polyvinyl acetate adhesive was purchased from Shanghai Zhongnan Building Materials Co., Ltd., Shanghai, China.

Bamboo was purchased from Zhejiang Yongyu Bamboo Co., Ltd., Huzhou, China, and cut into 15 bamboo strips of the same quality with dimensions of 100 mm × 20 mm × 5 mm with their longitudinal faces parallel to the direction of the grain, the process of obtaining bamboo strips is shown in Fig. 1.

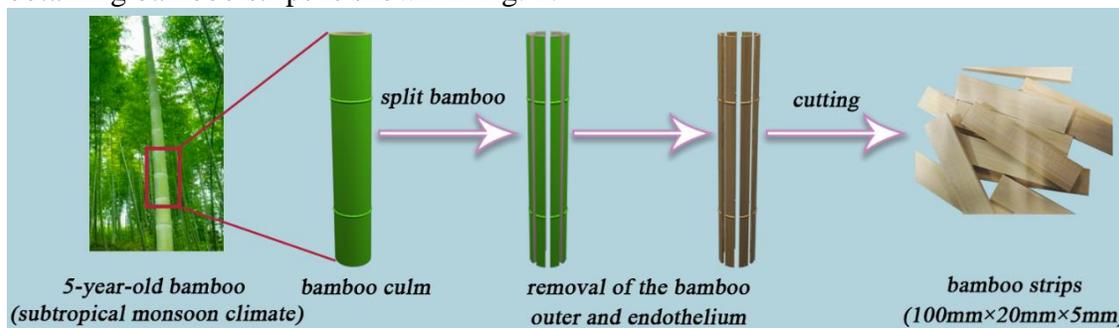


Fig. 1. Schematic diagram of the process of obtaining bamboo strips

Synthesis of the LDHs Fire Retardant

First, 16.923 g Mg(NO₃)₂, 8.253 g Al(NO₃)₃, and 35.676 g (NH₂)₂CO were added to the beaker, followed by the addition of 125 mL of deionized water, and the solution was clarified by stirring. Then, the clear solution was transferred to a three-necked flask and heated to 100 °C for 24 h. The temperature was kept constant at 100 °C until the mixture reached a static crystallization state for 10 h, after which the MgAl-LDHs powder was obtained by filtration, washing, drying, and grinding.

Retardancy Treatment of the MgAl-LDHs on Bamboo

For the MgAl-LDHs alone treatment, the aqueous solution of the MgAl-LDHs was first prepared into a suspension liquid of 1% by treatment with ultrasonic waves for 1 h at room temperature. Five bamboo strips were placed in the MgAl-LDHs suspension liquid and stirred to make sure that the MgAl-LDHs did not precipitate. Five pieces of bamboo were completely immersed in the MgAl-LDHs suspension liquid for 4 h at normal room temperature and pressure. The samples (named LB) were washed with deionized water and dried (Fig. 2a).

For the pretreatment with sodium hydroxide and MgAl-LDHs, five bamboo strips were first pretreated with sodium hydroxide. The 5% concentrated (mass concentration) sodium hydroxide solution was used to impregnate the bamboo for 30 min and was washed with deionized water until the pH reached 7. The MgAl-LDHs was prepared in a suspension liquid of 1% using ultrasonic waves for 1 h at room temperature. Then, the bamboo was immersed in a 1% MgAl-LDHs suspension for 4 hours at room temperature and pressure. The sample was washed with deionized water, dried, and named NLB (Fig. 2b).

The remaining five bamboo strips were untreated controls.

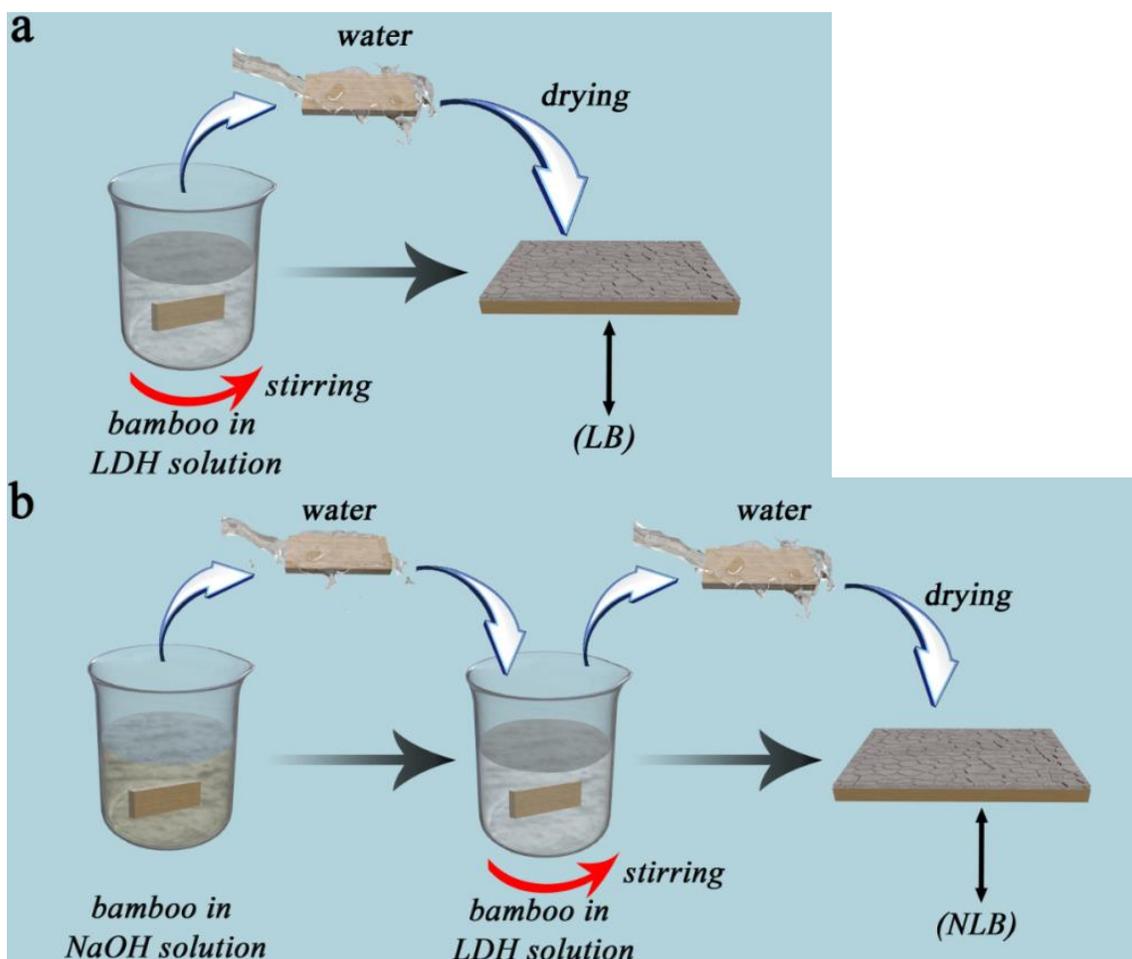


Fig. 2. Schematic diagram of preparation of the MgAl-LDHs coating on bamboo surface

Calculation Method of LB and NLB for Loading Dosage

The bamboo pieces were placed in an oven to be dried to have a water content of about 10%; the mass of the bamboo pieces was measured to be m_1 , and the bamboo was used to prepare LB and NLB. Next, LB and NLB were dried in an oven to have a water content of about 10%, and the mass of the bamboo piece was measured to be m_2 . The loading dosage is the percentage change of bamboo quality before and after flame retardant modification. The loading dosage can be calculated by the following formula,

$$\text{Loading dosage (\%)} = \frac{m_2 - m_1}{m_1} \times 100\% \quad (1)$$

Chemical Component Analysis

XPS analyses of samples of untreated bamboo, LB and NLB were carried out on an ESCALAB250 spectrometer (ThermoVG, USA) with a pass energy of 15 mA and AlK α radiation of 12 kV ($h\nu = 1486.7$ eV). Samples of untreated bamboo and sodium hydroxide treated bamboo were milled for FT-IR analysis; the mass ratio of bamboo and KBr was 1:100 and the scan range was 4000 to 400 cm^{-1} with 32 scans at 4 cm^{-1} resolution for each sample. The composition of the MgAl-LDHs was determined by an XRD-6000 device (SHIMADZU, Japan) equipped with a Cu K α radiation source ($\lambda = 0.154$ nm). The analyses were performed with an acceleration voltage of 20 kV and electric current of 30 mA. A 2 θ range was scanned between 5 $^\circ$ and 70 $^\circ$ in a fixed time mode at a step interval of 2 $^\circ$ /min.

Microscopy and Microanalysis Testing

Scanning electron microscopy (SEM) scans of untreated, LB, and NLB samples were obtained using a scanning electron microscope. Microanalysis was conducted using an energy-dispersive X-ray spectrometer (Genesis 4000, AMETEK, Newark, DE, USA). Samples were covered by galvanic gold and 25 kV acceleration voltage was applied for testing.

Cone Calorimeter Test

A 100-mm square specimen (dimensions 100 mm \times 100 mm \times 5 mm) was prepared using polyvinyl acetate adhesive and bamboo (Fig. 3). Cone calorimeter testing was performed following the ISO standard 5660-1 (ISO 1993) standards with a FTT0242 cone calorimeter (FTT Company, Derby, UK). The specimen was exposed to a constant, external heating flux and fixed by a stainless-steel mesh on the surface. All tests were conducted at an irradiance level of 50 kW/m^2 .

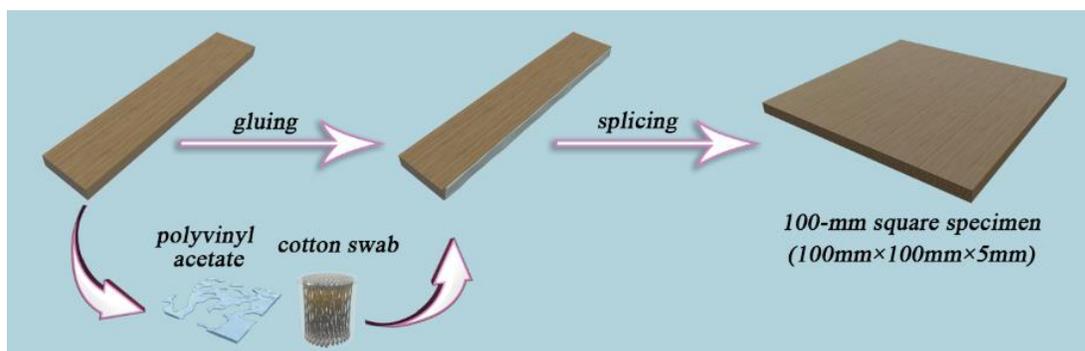


Fig. 3. Schematic diagram of combustion sample preparation

RESULTS AND DISCUSSION

Characterization of MgAl-LDHs

The XRD spectra of the MgAl-LDHs is shown in Fig. 4. Compared with that of the PDF#89-0460[(Mg_{0.667}Al_{0.333})(OH)₂(CO₃)_{0.167}(H₂O)_{0.5}] in the JADE6.0, the corresponding characteristic peaks appeared at 11.64°, 23.48°, 34.86°, 35.62°, 60.82°, and 62.14°.

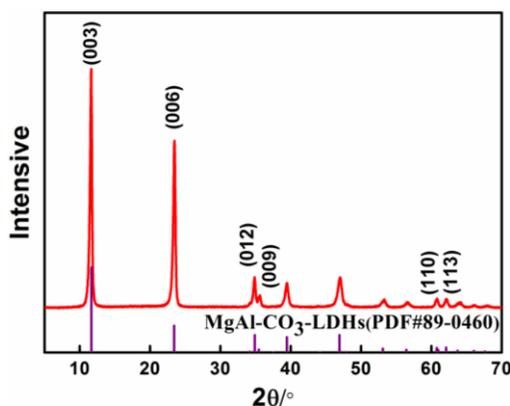


Fig. 4. XRD spectra of the MgAl-LDHs synthesized by urea hydrolysis method.

According to Bragg's equation, the diffraction peak spacing of the 003 peak was 0.76 nm (Fig. 4), illustrating that the interlaminal anions of the MgAl-LDHs synthesized in this study were carbonate ions (Violante *et al.* 2009). It is indicated that the MgAl-LDHs with interlayer anions being carbonate ions were successfully synthesized by urea hydrolysis method, and used as a flame retardant for bamboo.

Morphology and Chemical Bond State Analysis

The XPS spectra of the untreated, LB, and NLB bamboo samples are shown in Fig. 5. Figure 6 shows the digital photos and SEM images of untreated bamboo, LB, and NLB. The relative intensity of the peaks at C1s and O1s of the LB and NLB spectra are lower than that of untreated bamboo (Fig. 5). Additionally, the peaks at 1303.5 eV and 74.3 eV were changed to the binding energy of Mg1s and Al2p, which are the characteristic peaks of MgAl-LDHs. These changes indicated that the MgAl-LDHs fire retardant was successfully synthesized and applied to the surface of bamboo.

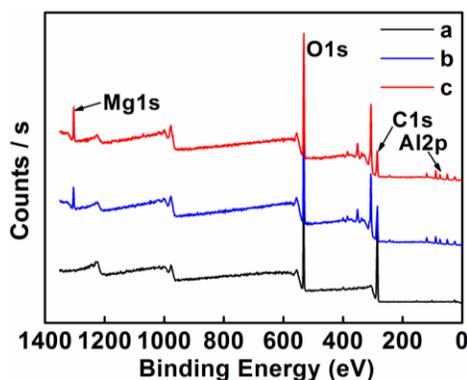


Fig. 5. XPS spectra of (a) untreated bamboo, (b) LB, and (c) NLB

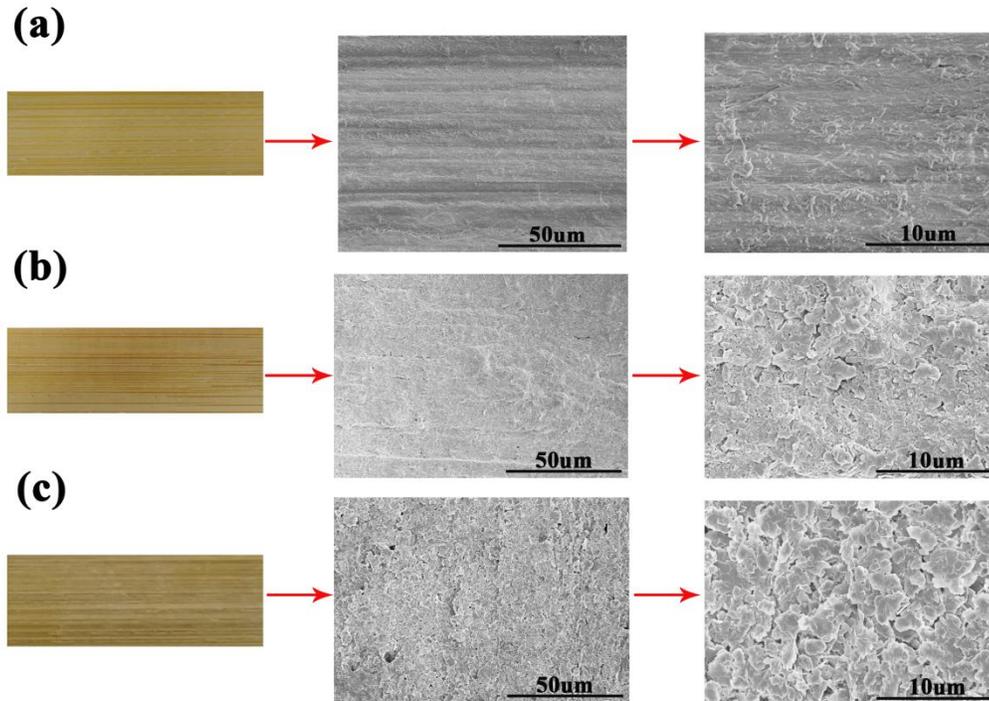


Fig. 6. Digital photographs and SEM images of untreated bamboo (a), LB (b), and NLB (c). Note: The high magnification images were all selected from same place as the previous image.

Figure 6b (LB) shows that a continuous coating of MgAl-LDHs was formed on the surface of bamboo after treatment with MgAl-LDHs suspension. Figure 6c (NLB) shows a sample pretreated with sodium hydroxide solution and then treated with MgAl-LDHs. It shows a denser layer of coating of MgAl-LDHs on the surface of bamboo, further proving that the MgAl-LDHs coating generated by this method was firmly connected with the bamboo surface. Some areas on the surface of bamboo appear light grey in the micrographs, and others appear dark grey; this can be explained by the distribution of the MgAl-LDHs on the surface. The MgAl-LDHs were gathered together and stacked on the surface, leading to an uneven distribution of MgAl-LDHs.

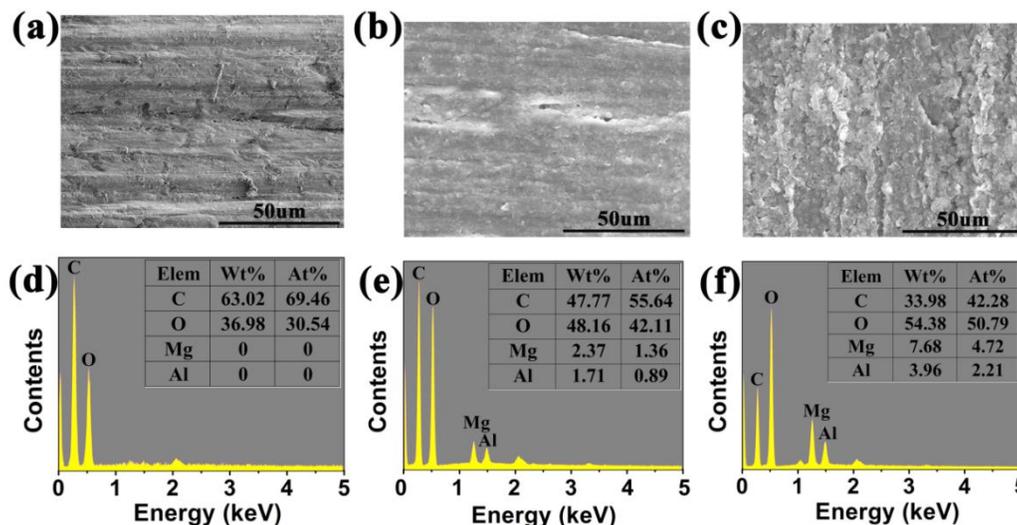


Fig. 7. EDX spectrum of the untreated bamboo (a and d), LB (b and e), and NLB (c and f)

The results of elemental analysis of each specimen are given in Fig. 7. The untreated bamboo and the treated bamboo pretreated with sodium-hydroxide solution (NLB) were analyzed by FT-IR, as shown in Fig. 8. Figure 7 shows that the content of magnesium and aluminum on the surface of NLB samples (Fig. 7f) was about three times higher than that of the group treated by MgAl-LDHs alone (LB, Fig. 7e). Because of the treatment with sodium hydroxide, more hydroxyl groups were exposed on the surface of bamboo and promoted the formation of the MgAl-LDHs coating.

To confirm that there was an increased number of hydroxyl groups on the sodium hydroxide treated bamboo, samples were analyzed by FT-IR (Fig. 8). Both the untreated bamboo and the sodium hydroxide treated bamboo showed a wide and large peak at 3427 cm^{-1} , which represented the asymmetric stretching vibration of the -OH group (Fu *et al.* 2012). However, the intensity of the peak was stronger than that of untreated bamboo, revealing that the surface of bamboo possessed more hydroxyl groups after being treated with sodium hydroxide. This result was consistent with a previous report (Jia *et al.* 2018). Thus, the increased amount of hydroxyl groups on the surface of the sodium hydroxide treated bamboo was due to the treatment of sodium hydroxide solution, which facilitated the formation of hydrogen bonds between the MgAl-LDHs and the bamboo surface and fixed the MgAl-LDHs fire retardant on the surface.

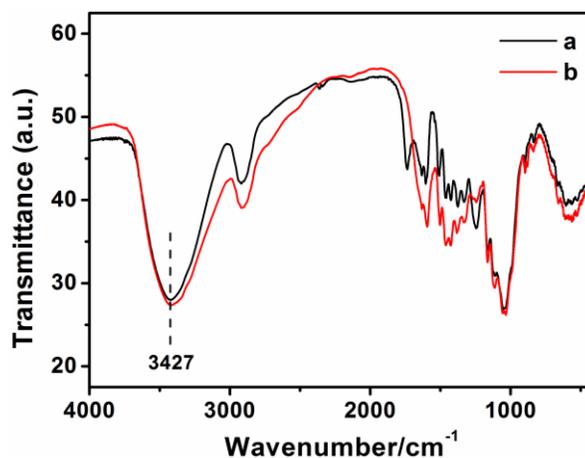


Fig. 8. FT-IR spectra of untreated bamboo (a) and the treated bamboo pretreated with sodium-hydroxide solution (b)

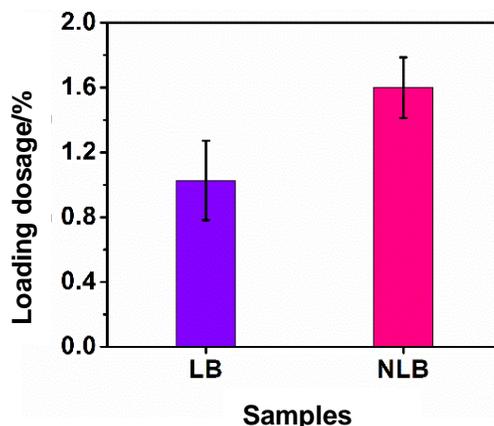


Fig. 9. Loading dosage of samples

Loading Dosage of LB and NLB

To further understand the MgAl-LDHs loading in LB and NLB, this study calculated the loading dosage of LB and NLB. Figure 9 shows the loading dosage of LB and NLB. As can be seen from Fig. 9, the loading dosage of NLB is 56.9% more than the loading dosage of LB. The result is consistent with the results obtained in Fig. 6 and Fig. 7. Therefore, this may further indicate that the bamboo treated with NaOH can load more MgAl-LDHs.

Flammability Testing

The flammability of untreated NLB bamboo was tested using the Cone. The heat release rate (HRR), the total heat release (THR), smoke release rate (SPR), the total smoke production (TSP), carbon monoxide production (COP), and the average rate of heat emission (ARHE) were measured. The results are shown in Table 1 and Fig. 10.

Table 1. Heat Release Properties of Untreated and Treated Bamboo

Sample code	TTI (s)	PHRR (KW/m ²)	TPHRR (s)	AEHC (MJ/kg)
Untreated bamboo	16	525.4	110.2	12.4
NLB	28	372.4	128.7	10.5

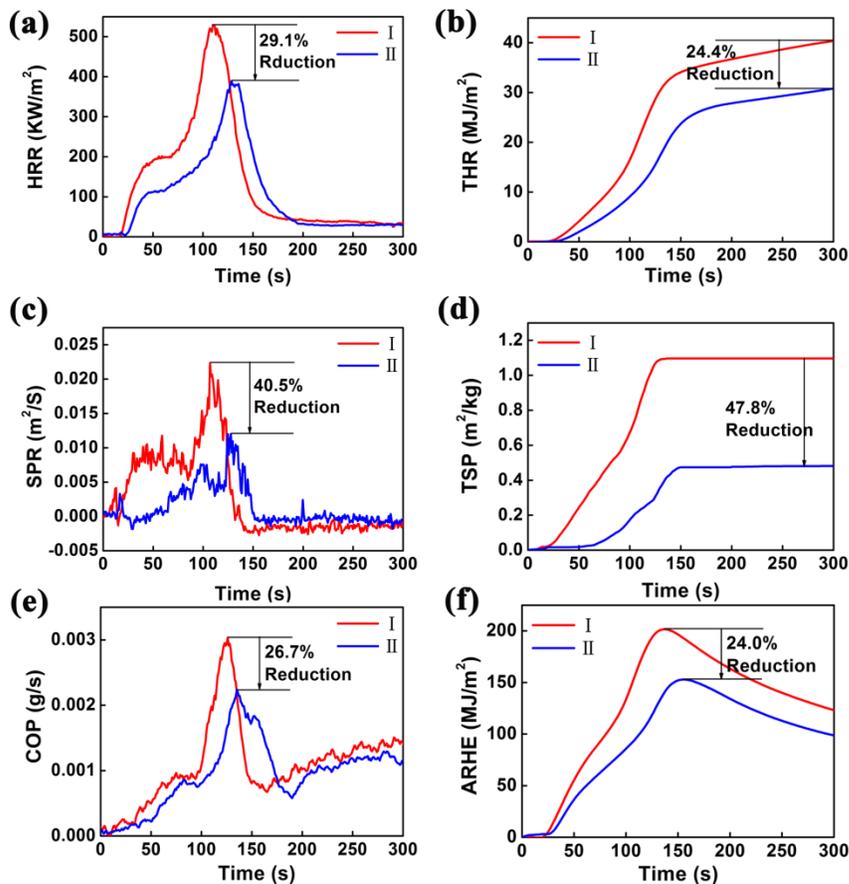


Fig. 10. Cone of untreated bamboo (I) and the NLB (II): heat release rate (a), total heat release rate (b), smoke release rate (c), total smoke release rate (d), carbon monoxide production rate (e), and average rate of heat emission (f)

Generally, a greater maximum value of the HRR results in a greater fire risk for the material. Conversely, a smaller HRR value leads to better fire retardancy (Du *et al.* 2017a). To enhance the fire retardancy of treated bamboo, the HRR peak value should be reduced as much as possible.

As shown in Fig. 7a and Table 1, the peak HRR value (PHRR) of untreated bamboo was 525.4 kw/m², while the PHRR value of the NLB was 372.4 kw/m². This represents a decrease of 29.1% compared with that of the untreated bamboo. Ignition time (TTI) and time of the HRR peak (TPHRR) of NLB was delayed by 75% and 16.8% compared with untreated samples, respectively. Hence, the fire retardancy of the NLB was improved by treatment with MgAl-LDH fire retardant. The MgAl-LDH fire retardant absorbed heat and produced intermediate products like water and CO₂, which diluted the oxygen content during the thermal degradation process. The thermal decomposition of the MgAl-LDH fire retardant decreased the heat release rate and increased the TTI and TTHR (Elbasuney 2015; Guo *et al.* 2017).

The total heat release (THR) curves of the untreated bamboo and the NLB are shown in Fig. 10b. The THR curve of the NLB increased with the elapse of time, but the curve was lower than that of untreated bamboo during the entire testing process. This result was also consistent with that of the HRR curves. According to the Fig. 10b and Table 1, the total heat release of the NLB was 24.4% lower than that of untreated bamboo. The combustion of the NLB was effectively inhibited by the coating of the MgAl-LDHs fire retardant, thus reducing the total amount of heat released.

The generation of the smoke and poison gas from the burning of wooden materials is even more harmful than the combustion itself. It is one of the main causes of casualties during fires (Du *et al.* 2017b). It is necessary to detect the degree of smoke released and poisonous gas production during the combustion process. According to Figs. 7c and d, the SPR of the NLB was 40.5% lower than that of untreated bamboo. Fig. 10d shows the TSP curves of untreated bamboo and the NLB; the TSP had a decrease of 47.8% compared with that of untreated bamboo. The NLB showed excellent smoke suppression properties because of the MgAl-LDH coating. This phenomenon can be explained by the physical barrier on the bamboo surface provided by the MgAl-LDH fire retardant, which inhibited the conversion action of organic macromolecules into organic gas volatiles during the combustion process. In addition, the thermal decomposition of the MgAl-LDHs fire retardant created alkaline metal oxides, which absorb some intermediate acidic gases products, further reducing the total amount of smoke released (Zammarano *et al.* 2005).

According to Table 1, the average effective combustion heat (AEHC) of the NLB was 15.7% lower than that of untreated bamboo, which further indicates the reduced amount of releases of volatile gas products in the combustion process. The peak value of the CO production was 26.7% lower than that of untreated bamboo (Fig. 10e). Therefore, the smoke release and toxic gas (namely CO) produced by the combustion process of the NLB were decreased. During a fire, the MgAl-LDH coating of this modified bamboo could delay the spread of fire and help provide more time for evacuation, thus potentially reducing the number of casualties.

Fig. 10f displays the average rate of heat emission (ARHE) curves of untreated bamboo and NLB. The maximum average rate of heat emission (MARHE) can be used as an index for measuring the development of fires in real scenarios (Sacristán *et al.* 2010). The MARHE value of untreated bamboo was 202.2 MJ/m², while the MARHE value of NLB was only 153.6 MJ/m², which confirmed the improved fire retardancy of the NLB after being treated by the MgAl-LDHs fire retardant.

The digital photos of the residual charcoal of untreated bamboo and the NLB after combustion are shown in Fig. 11. A large amount of ash was generated after the burning of untreated bamboo. The original size of the bamboo was greatly reduced, and some serious deformation and fragmentation was found on the surface of the remains. In contrast, the residual charcoal of the NLB maintained the original size and shape of the bamboo after the combustion process. Instead of the ash products, the surface of charcoal from the NLB was covered with a white inorganic layer. This means that the MgAl-LDH flame retardant can effectively promote the production of charcoal. The bamboo treated with both sodium hydroxide solution and MgAl-LDH fire retardant (namely NLB) had good fire retardancy properties.



Fig. 11. Digital photographs of the residual charcoal: untreated bamboo (a), NLB (b)

CONCLUSIONS

1. Pretreatment of bamboo with sodium hydroxide solution can increase the amount of hydroxyl groups exposed on the surface of bamboo, thus enhancing the formation of hydrogen bonds between the MgAl-LDHs and the bamboo. The MgAl-LDH coating on the surface of bamboo became stronger and denser, which further increased the leaching resistance of this fire retardant.
2. Cone calorimeter analysis showed that the two-step method for fabricating bamboo coated with MgAl-LDHs (NLB) has excellent flame retardant properties. When compared with that of untreated bamboo, the peak of heat release rate (PHRR) and total heat release (THR) of NLB decreased by 29.1% and 24.4%, respectively; the smoke release rate (SPR) and total smoke release (TSP) decreased by 40.5% and 47.8%, respectively; the peak of the carbon monoxide production rate (PCOP) decreased by 26.7%; and the maximum average rate of heat emission (MARHE) decreased by 24.0%.
3. The fire retardant and smoke suppression properties of the bamboo were greatly improved by the two-step method using the MgAl-LDHs fire retardant. At the same time, the MgAl-LDHs coating formed on the surface of bamboo was more stable and less leaching. The MgAl-LDHs used in this study is a kind of high efficiency inorganic fire retardant that can be applied to the bamboo industry.

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