Effects of Boric Acid and/or Borax Treatments on the Fire Resistance of Bamboo Filament

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Bamboo filament, a material often used for indoor decoration, should be treated with flame retardants for safe use. This study evaluated the effects of different boron fire retardants on the heat release and smoke release of bamboo filaments and untreated samples *via* a cone analysis. A thermogravimetric analyzer (TA) instrument was used to investigate the fire retardant mechanisms of the different boron compounds. The results showed that compared to the untreated samples, fire retardants that contained boric acid or borax effectively reduced the heat and smoke release from the bamboo filament. The effects of the different ingredients in the fire retardant on the combustion process were quite different. During the combustion process, borax displayed better performance for restraining the heat release rate than boric acid, while for the total amount of heat release and the smoke suspension performance, the result was the converse. The excellent synergistic effect could be obtained by a mixture that contained a reasonable proportion of boric acid and borax (Boric Acid:Borax = 1:1). In the pyrolysis process, boric acid had stronger catalytic dehydration, while the mass loss in the treated samples with boric acid or higher proportions of boric acid was less than the loss in the borax-treated samples.

Keywords: Bamboo filament; Boric acid; Borax; Heat release; Smoke release; TG analysis

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INTRODUCTION

As a natural raw material, bamboo is an abundant natural resource in Asia and South America, especially in China, where the total area of bamboo is about six million hectares (Atanda 2015; Liu *et al.* 2016). Nowadays bamboo is a promising substitute material for flooring, roofing, chopsticks, paper-making, biomass solid fuel, and reinforcement in place of steel in buildings due to its innate properties. These properties include its low density, high tensile modulus, environmental friendliness, low cost, and local accessibility (Zhang *et al.* 2010; Zakikhani *et al.* 2014). The superior mechanical properties of bamboo are mainly attributed to its unidirectional oriented fibers that account for 40% of the culm by volume (Zou *et al.* 2009; Fei *et al.* 2016). Moreover, bamboo only takes several months to grow (Okubo *et al.* 2004), whereas wood needs more than ten years. This is important for some developing countries, such as China, because natural forestry resources have been strictly prevented in cutting down trees in broad scale and the demand gap for wood raw material has gradually expanded in recent years.

Among bamboo-based products, bamboo filament is becoming popular as an indoor decoration material that can substitute traditional wallpaper or paint in house refurbishment because it can be applied to the wall quickly, removed, or reused conveniently, and the application process is environmentally safe (Yu *et al.* 2017). However, as with other forest species, the biggest challenge for the safe use of bamboo filament is its innate inflammable property. According to the compulsory Chinese national standards GB/T 20286 (2006) and GB/T 8624 (2006) issued by the Fire Department of the Chinese Ministry of public security, construction and indoor decoration materials must be treated with suitable fire retardants to reach a nonflammable level. To reduce flammability and guarantee their safe use, research has been widely conducted to improve the fire resistance of bamboo-based materials. Jin *et al.* (2016) treated moso bamboo strips with compound flame retardants including ammonium polyphosphate (APP), diammonium phosphate (DAP), monoammonium phosphate (MAP), and boric acid/borax. Compared with untreated bamboo, the initial thermal degradation temperature decreased sharply, the char yield increased from 58% to 74% in the treated bamboo, and the total smoke release and the total heat release were reduced 48.49% and 84.92%, respectively. Xu and Li (2013) mixed ammonium hydrogen, phosphate, ammonium sulfate, ammonium polyphosphate, and borax (30:35:8:27) to treat sliced bamboo plywood, and they found that compared with the untreated bamboo plywood, although the bonding strength of the fire-retardant plywood decreased, the heat release ratio and total heat release were greatly reduced. The effect of the fire retardant was significant; the peak smoke release rates of the fire-retardant plywood decreased 38.93%; total smoke release decreased 44.38%. Other compound flame retardants with guanylurea polyphosphate (GUP) and boric acid also have achieved better fire resistant performance in treated bamboo (Zhu and Huang 2009).

In frequently used compound fire retardants, boron components have only been used as a booster due to poor leachability (Toussaint *et al.* 2000; Ramos *et al.* 2006; Yamauchi *et al.* 2007). However, fire retardant systems containing boron have attracted attention for indoor decoration material treatment because they are halogen-free, colorless, odorless, inexpensive, low in mammalian toxicity, and of low volatility (Zhang *et al.* 2015; Liu 2016). The effectiveness of many boron compounds (*e.g.*, boric acid, borax, *etc*.) used as fire retardants in wood and bamboo-based materials has been widely reported (Jiang *et al.* 2011; Kurt *et al.* 2012; Dogan 2014; Tondic *et al.* 2014; Cavda *et al.* 2015; Zhang *et al.* 2015; Liu 2016). Borax tends to reduce the flame spread, but it can promote smoldering or glowing. In contrast, boric acid suppresses glowing but has little effect on the flame spread. Boric acid catalyzes the dehydration and other oxygen-eliminating reactions of wood at a relatively low temperature (100 °C to 300 °C). It may catalyze the isomerization of the newly formed polymeric materials to form aromatic structures. This contributes partly to the effects of boric acid promoting charring and the fire retardation of wood. In the authors' previous research, it was found that compared to the untreated bamboo filaments, heat and smoke release could be greatly reduced in the treated bamboo filaments with a higher concentration of boric acid and borax mixture (1:1), and during the combustion process, the mixture of boric acid and borax fire retardants could promote the carbon residue production of the treated bamboo filament, thus reducing the mass loss (Yu *et al.* 2017).

The purpose of this study was to further investigate the effects of different boric acid and/or borax treatments on the thermal curing behavior of bamboo filaments, including combustion performance, and the difference of fire retardant mechanisms in the boric acid and/or borax system.

EXPERIMENTAL

Materials

Samples and treatment

Moso bamboo (*Phyllostachys edulis* (Carr.) H.de Lehaie) was taken from Hubei Province, China. After being air-dried, the moso bamboo was cut along the direction of the fiber to obtain several bamboo splits, then the bamboo filaments were drawn from the bamboo splits by a drawing machine (XinyiManufacturer Corporation, Nanning, China). The filaments were stuck together with non-woven fabric by a polyvinyl acetate adhesive and cut into small pieces with dimensions of 100 mm (tangential) × 100 mm (longitudinal) × 1.5 mm (radial). Bamboo filaments with similar weights were selected as the test samples. The components for boric acid and/or borax fire retardants and treatment conditions are listed in Table 1, and three replicates in the same treatment conditions. For T1 group, the mass fraction of boric acid and borax was 10%, respectively, which meant in 100 g T1 solution, 10 g boric acid and 10 g borax were dissolved in 80 g water. For the other groups, the same preparation method was used according to the mass fraction of different ingredients. The filaments were immersed into 100 °C boric/and borax solutions with different concentrations for 2 h, and then the filaments were removed into the conditioning room at 50°C and R.H. 60% to reach constant weight.

Table 1. Formulas of Boric Acid and/or Borax Fire Retardants

<table>
<tr><th>Sample Label</th><th>Components</th><th>Concentrations (%)</th><th>Treatment Temperature (℃)</th><th>Treatment Duration (h)</th><th>Replicates</th></tr>
<tr><td>Untreated</td><td>-</td><td>-</td><td>-</td><td>-</td><td rowspan="6">6</td></tr>
<tr><td>T1</td><td>Boric Acid:Borax = 1:1</td><td>20</td><td rowspan="5">100</td><td rowspan="5">2</td></tr>
<tr><td>T2</td><td>Boric Acid</td><td>20</td></tr>
<tr><td>T3</td><td>Borax</td><td>20</td></tr>
<tr><td>T4</td><td>Boric Acid:Borax = 7:3</td><td>20</td></tr>
<tr><td>T5</td><td>Boric Acid:Borax = 3:7</td><td>20</td></tr>
</table>

Methods

Combustion performance by cone calorimeter

After conditioning, the bamboo filaments were dried at room temperature. One group of bamboo filaments without any treatment was used for reference. The combustion performance of the specimens with different boric acid and/or borax fire retardant treatments was evaluated by a cone calorimeter according to ISO 5660 (2002). Six specimens with the same treatment condition were prepared with dimensions of 100 mm × 100 mm × 1.5 mm, and placed horizontally under a cone heater with a heat flux of 50 kW/m^2. A stainless steel cover with an opening of 0.0088 m^2 on the upper part was attached. The data was recorded by a computer on a second by second basis.

Thermal decomposition

The thermal decomposition characteristics were analyzed using a TA instrument Q 500 (TA Instrument Corporation, Wilmington, USA). Thermogravimetric analysis (TGA) is a technique that provides quantitative decomposition information on a polymeric material and can be used to study degradation kinetics and char formation.

The samples were milled to 80-mesh powder and oven-dried. Approximately 5 mg to 8 mg of the sample powder was evenly and loosely distributed in an open pan. Temperature variation was controlled from room temperature to 800 °C with a heating rate of 10 °C /min. In this study, air was used as the reactive gas at a constant flow of 10 mL/min, and nitrogen was used as the protective gas also at a constant flow of 10 mL/min. Weight loss of the samples was continuously recorded during the thermal decomposition process. Three replicates of each TGA experiment were performed. The experimental data could be directly obtained through the TGA Q 500 thermogravimetric analyzer; the data were analyzed using universal analysis software from TA instruments (Universal Analysis 2000, Wilmington, America).

Statistical analysis

The effects of different treatments on the smoke generation parameters of the treated bamboo filaments were evaluated by one-way analysis of variance and S-N-K(S) test calculated in the SPSS software. In the variance analysis, the significant value P was calculated and compared with the critical value 0.05. $P < 0.05$ means: the factor has a significant effect on the experimental result.

RESULTS AND DISCUSSION

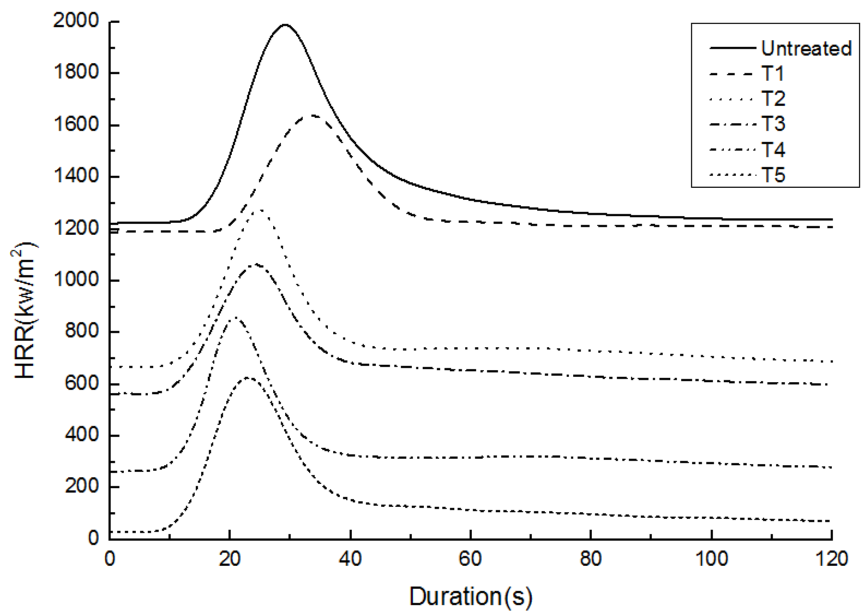
Heat Release Analysis

The heat release rates (HRR) for the bamboo filaments with different fire retardant treatments are shown in Fig. 1(a), and the corresponding peak HRR (pkHRR) values are shown in Table 2. These two parameters are important to describe the pyrolysis and flame spread speed during the combustion process.

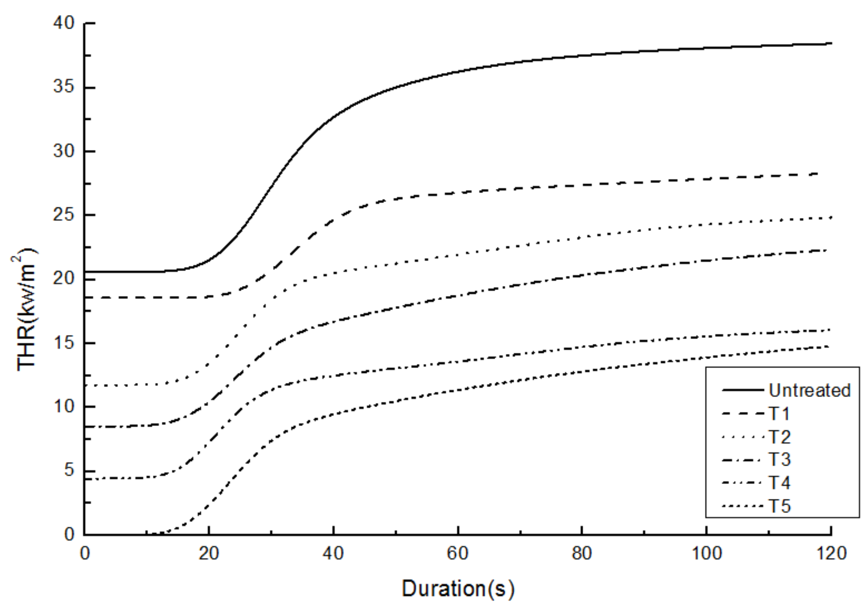
From Fig. 1(a), during the whole combustion process, the HRR of the samples with different treatments exhibited some special characteristics: in the first 25 s, except for the samples of T1 (Boric Acid:Borax = 1:1), the HRR of the untreated samples were slower than the other four groups of the treated samples; however, the results from 25 s to 60 s were reversed in that the HRR of the untreated samples were much faster than all of the treated samples.

After 60 s, the HRR of all groups were reduced to a similar lower level. For the time it takes the samples to peak (Table 2), it can be observed that the heat release process was only delayed in group T1 compared to the untreated group. For the values of pkHRR, the lowest value was also observed in T1 samples, which was reduced by 40% compared to the untreated samples.

For the samples treated with different proportions of boric acid and borax, the order of the pkHRR values was: T1 < T3 (Borax) < T5 (Boric Acid:Borax = 3:7) < T2 (Boric Acid) < T4 (Boric Acid:Borax = 7:3). Borax can be seen as showing better performance for restraining the heat release than boric acid, and an excellent synergistic effect could be obtained by a mixture of boric acid and borax with a reasonable proportion (Boric Acid:Borax = 1:1).



(a) Heat release rate (HRR)



(b) Total amount of heat release (THR)

Fig. 1. Effects of different fire retardant treatments on the heat release of bamboo filaments

Table 2. Peak of HRR from the Treated Bamboo Filament with Different Treatments

Sample Label	Un-treated	SD	T1	SD	T2	SD	T3	SD	T4	SD	T5	SD
Mean pkHRR (kW·m^2)	843.50	1.29	482.10	1.53	717.90	1.12	565.20	2.13	729.44	1.53	615.30	1.63
Mean Time to Peak (s)	30	1.87	35	1.03	25	1.42	25	1.76	20	2.07	25	1.39

The total amounts of heat release (THR) for the bamboo filaments with different fire retardant treatments are shown in Fig. 1(b). It can be observed that although all the treatments played effective roles in reducing the THR values, the samples' values in T1 were much less than those in the other groups, and compared to the untreated samples, it could be reduced by 46.1%. For the samples treated with different proportions of boric acid and borax, the order of the THR values was: T1 < T4 (Boric Acid:Borax = 7:3) < T2 (Boric Acid) < T3 (Borax) < T5 (Boric Acid:Borax = 3:7). This result demonstrated that boric acid had better performance in restraining the amount of heat release than borax, and an excellent synergistic effect could also be obtained by the mixture of boric acid and borax with the reasonable proportion (Boric Acid:Borax = 1:1).

Those results were attributed to the different fire retardant mechanisms of boric acid and borax. On the one hand, these two compounds have the same commonly accepted mechanism that a coating or protective layer is formed on the wood surface at higher temperatures (Kandola and Horrocks 1996). In contrast, some researchers have established that borax tends to reduce flame spread, but it can promote smoldering or glowing; while in other instances, boric acid suppresses glowing but has little effect on the flame spread (Baysal 1994; Hafizoglu *et al.* 1994). Wang *et al.* (2004) also found that boric acid could catalyze the dehydration and other oxygen-eliminating reactions of wood at a relatively low temperature (approximately 100 °C to 300 °C) and may catalyze the isomerization of the newly formed polymeric materials by forming aromatic structures. This result partly contributes to the effect of boric acid on promoting the charring and fire retardation of wood. Thus, for boric acid treatments on bamboo filaments, because of the smaller thickness and bigger void ratio of the filaments, they were more prone to promote the flame spread, but restrained the amount of heat release. The results have shown that when the proportion between boric acid and borax was 1:1, the heat release could be effectively retarded in the treated samples. This was probably attributable to the more stable and effective chemical components produced by this seasonable proportion in the treated bamboo filaments.

The ignition time and flameout time are shown in Table 3. It seemed that compared to the untreated samples, the ignition time of bamboo filaments with different fire retardant treatments could be delayed, and the flameout time could be shortened in various degrees. The results also demonstrated that borax tends to reduce flame spread, but it can promote smoldering or glowing; while in other instances, boric acid suppresses glowing but has little effect on the flame spread. The ideal proportion between boric acid and borax was 1:1, which could delay the ignition time the most effectively and suppress the flame spread obviously.

Table 3. Ignite Time and Flameout Time of the Treated Bamboo Filament with Different Treatments

Sample Label	Un-treated	SD	T1	SD	T2	SD	T3	SD	T4	SD	T5	SD
Mean Ignite Time (s)	9	1.55	17	1.34	21	1.15	10	2.04	12	0.87	10	2.23
Mean Flameout Time (s)	84	2.79	56	1.09	80	2.59	52	1.30	78	1.64	81	1.46

Smoke and Gas Release Analysis

The total amount of smoke release per unit (TSP) of the material during the fire and the average amount of smoke release from per mass (ASEA) of the material during the fire are shown in Fig. 2 and Table 3. Compared to the untreated samples, the TSP and ASEA were reduced dramatically in the boron-treated bamboo filaments. The minimum TSP was also found in the T1 group samples. Compared to the untreated samples, the values of TSP and ASEA could be reduced by 91.81% and 87.92%, respectively. The excellent smoke suppression of boron compound fire retardants has also been established by past research (Wang 2000; Mukherjee and Ansuman 2002), in which boron compounds were chosen to reduce the smoke release in some fire retardant formulas.

The reasons for the better smoke suppression appear to involve two aspects: one is that boric acid and borax mixtures have some efficacy in a char-forming catalytic effect at a relatively lower temperature (Wang *et al.* 2004); the other is that they also have a rather lower melting point and form glassy films when exposed to higher temperatures in fires (Nussbaum 1988).

From Fig. 2, it can be observed that the proportions between boric acid and borax had an important effect on the smoke release, and the excellent synergistic effect could be also achieved by the mixture of boric acid and borax with a reasonable proportion (T1-Boric Acid:Borax = 1:1). It seemed that boric acid had better smoke suspension performance than borax, for the same reasons previously mentioned in the results of the amount of heat release.

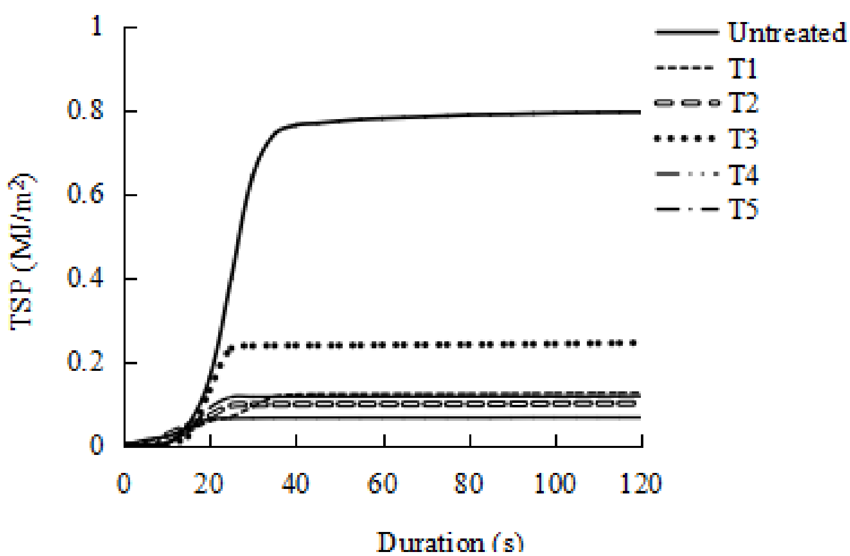


Fig. 2. Effects of different fire retardant treatments on the smoke release of the bamboo filaments

In Table 3, more CO and CO_2 were produced from the untreated bamboo filaments during the combustion process. All the average CO and CO_2 productions from the treated samples, especially for the samples in the T1 group, experienced reduction. The results demonstrated that boric acid/or borax compounds aided in the reduction of the toxic gas production, especially in reducing the insufficient combustion and decreasing the CO production.

Table 3. Smoke Generation Parameters of the Treated Bamboo Filament with Different Treatments

Sample Label	Untreated	T1	T2	T3	T4	T5
ASEA ($m^2 \cdot kg^{-1}$)	120.90	14.60	12.70	42.20	6.70	18.80
Average CO Production($kg \cdot kg^{-1}$)	0.11	0.09	0.07	0.04	0.09	0.05
Average CO_2 Production($kg \cdot kg^{-1}$)	2.23	1.65	1.84	2.03	1.89	2.05

The one-way analysis of variance for the treated bamboo filament with different formulas of fire retardants is shown in Table 4. It was noted that different treatment conditions had significant effects on the average amount of smoke release from per mass (ASEA), CO, and CO_2 production. The results indicated that bamboo filament treated with seasonable fire retardant was necessary for its safe application.

Table 4. One-way Analysis of Variance for the Treated Bamboo Filament in Different Treatment Conditions by SPSS

Quadratic Sum		df	F	Significant
ASEA	29811.81	17	1277.08	0.00
Average CO Production	0.012	17	21.30	0.00
Average CO_2 Production	0.603	17	1202.90	0.00

Thermal Decomposition Analysis of Bamboo Filament

Figure 3 shows the pyrolysis process of bamboo filaments with different fire retardant treatments at a heating rate of 10 °C/min in the air environment. The approximate start and end of the derivative thermogravimetric (DTG) curve shows thermal breakdown of organic matters in the samples, representing several activities during the course of combustion (Idris *et al.* 2013). The pyrolysis process for bamboo filaments included three stages: dehydration, oxidative pyrolysis, and char combustion, although between the last two stages there was no clear boundary. In the dehydration stage (below 200 °C), free water and bound water were evaporated from bamboo filaments. Compared to the untreated samples, maximum mass loss was observed in the boric acid treated samples (T2), while little mass loss was observed in the borax treated samples (T3), which was attributed to the catalytic dehydration of boric acid (Wang *et al.* 2004). The DTG curves of all the groups differed greatly in the last two stages, which indicated that the effects of different fire retardant treatments in the oxidation pyrolysis stage and char combustion stage were more noticeable than in the dehydration stage. The maximum pyrolysis peak temperature of the untreated samples in the oxidation pyrolysis stage was 289.3 °C, and it was lower than the treated samples. Moreover, the pyrolysis range for the untreated samples was bigger than the treated samples. These results confirmed the boron compounds effectively prevented the diffusion of heat and oxygen so that the pyrolysis process could be effectively retarded. These results were consistent with other research that the boron compound property could form a molten B_2O_3 film at a higher temperature, which could spread on the surface of the materials and effectively isolate the oxygen and fire (Yang and Qing 2014). Additionally, the boron compounds could release the bound water that cool the materials and absorb the heat during fire (Marosi *et al.* 2002; Silvo *et al.* 2007). The char combustion stage (above 400 °C), differed from other groups of the samples. For untreated wood and borax-treated wood (T3), a second pyrolysis peak could clearly be observed at 373.9 °C and 397.5 °C, respectively, which signified fast thermal decomposition in this process. This result also demonstrated that borax tended to promote smoldering or glowing, and boric acid suppressed glowing. As a result, the mass loss was over 90% for these samples, while the mass loss in the treated samples with boric acid or a higher proportion of boric acid could be reduced to less than 80%.

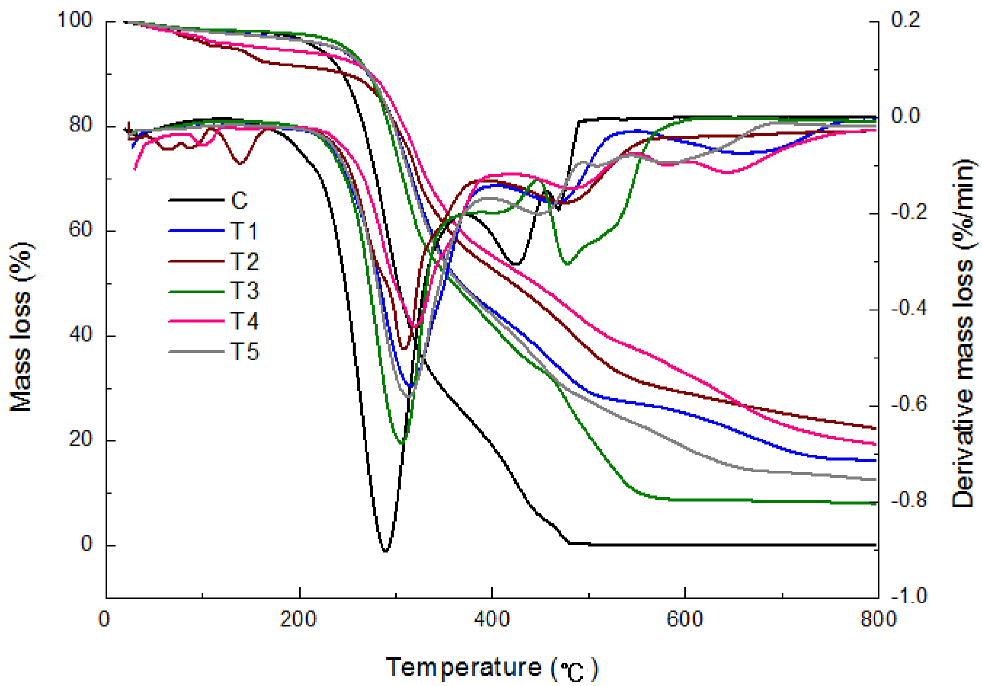


Fig. 3. Pyrolysis process of bamboo filaments with different fire retardant treatments in TG and DTG curves

CONCLUSIONS

1. Compared to the untreated samples, the effects of the ingredients in the boric acid and/or borax formulas were quite different during the combustion process. Borax displayed better performance for restraining the heat release rate than boric acid, while for the total amount of heat release, the result was *vice versa*. The excellent synergistic effect could be achieved by the mixture of boric acid and borax with the reasonable proportion (Boric Acid:Borax = 1:1).
2. This result contributed partly to the effect of boric acid on promoting charring and fire retardation of wood. Partly due to a smaller thickness and bigger void ratio of the bamboo filament, it was more prone to promote the flame spread but restrained the amount of heat release. When the proportion between boric acid and borax was 1:1, the heat release could be effectively retarded in the treated samples. This was probably attributable to the more stable and effective chemical components produced in this optimal proportion in the treated bamboo filaments.

3. Compared to the untreated samples, the TSP and ASEA were reduced dramatically in all the boron-treated bamboo filaments during the combustion process. Boric acid had better smoke suspension performance than borax. The average CO and CO_2 productions from the treated samples, especially for the T1 group samples, were noticeably reduced. This demonstrated that boric acid/or borax compounds aided in reducing the toxic gas production, especially in reducing the insufficient combustion and decreasing the CO production.
4. In the pyrolysis process, boric acid had stronger catalytic dehydration, while the mass loss in the treated samples with boric acid or higher proportion of boric acid was less than that in the borax-treated samples.

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