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STUDY ON THE MOST EFFECTIVE COMBINATION OF FLAME RETARDANT AND ANTI-AGING AGENT FOR BAMBOO MODIFICATION

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ABSTRACT

In order to promote the universal application of bamboo materials as well as to provide reference for the study of the performance of bamboo, four kinds of flame retardants (boric acid (BA), borax (BX), ammonium polyphosphate (APP), disodium octaborate tetrahydrate (DOT)), two kinds of ultraviolet light absorbers (UV-531, nano TiO_2) were added into waterborne polyurethane (WPU) to synthesize 5 kinds of modified coatings and coated on the surface of bamboo to make test materials. The flame-retardant analysis of the coated samples comprehensively explored the changes in flame retardancy of the test material after treatment. And the anti-aging test was carried out to investigate the color difference and adhesion change of the coating sample. The results show that the optimum flame retardant and anti-aging coating composition was obtained as 20% solid content of WPU, 8g coating amount of modifier, with the mass ratio of BA/BX/APP /DOT/water being 75:75:7:42:600, and the amount of UV-531 added being 1%.

KEYWORDS: Bamboo, water-based coating, flame retardant modification, anti-aging modification.

INTRODUCTION

Global ecological resources are being depleted, and the trend is getting worse. Forest resources, as the backbone of carbon sequestration, have also been seriously damaged in recent years, especially in some relatively resource-poor regions or countries (Martinho 2022, Mathis et al. 2021, Muhammad et al. 2023). Bamboo has excellent mechanical properties, short growth cycle, straight texture, renewable and other advantages, is considered an ideal alternative to wood resources (Chen et al. 2022, Hu et al. 2022, Li et al. 2022, Lian et al. 2023). However, bamboo also has its drawbacks, such as its surface often due to outdoor use, subjected to complex climatic conditions such as oxygen, ultraviolet radiation and various aging problems. Fire is easily caused by flame and heat radiation, which seriously affects the normal use of bamboo materials (Hu et al. 2021, Silvia et al. 2022, Zheng et al. 2019).

Boron-based and phosphorus-based flame retardants can provide good flame retardancy and mildew resistance to bamboo materials, not only have little effect on product performance, but also long-lasting flame-retardant effect (Cheng et al. 2020, Mestry and Mhaske 2019). Anti-aging additive UV-531 is a high-performance additive with light color, non-toxicity, and good compatibility for easy processing. It helps to delay the discoloration and loss of physical properties of bamboo and can provide good light stabilization for polyurethane coatings, etc., as well as good protection for plant fibers such as bamboo and wood (Lucas et al. 2021, Wang et al. 2023). Other anti-aging additives, such as ZnO and nano-TiO_2, can achieve a similar effect (Quintana et al. 2023, Tang et al. 2022, Xu et al. 2023). Conventional ways of introducing flame retardants and anti-aging agents are by impregnation treatments, but bamboo does not have radial distributed thin-walled cells, but rather contains abundant non-cell-wall materials such as colloidal substances, making it difficult to fully introduce the modified solution into the bamboo-based material (Chen et al. 2021, Li et al. 2023, Yuan et al. 2022). Waterborne polyurethane coatings, which use water as a solvent and produce coatings with excellent mechanical properties after application, have been widely used as protective coatings for bamboo utensils, and applying the modified coatings to the surface of bamboo ensures the uniformity of the introduction of the modifiers and saves costs while applying the protective coatings to the surface only (Chen et al. 2019, Mucci et al. 2023).

In this study, four flame retardants (boric acid (BA), borax (BX), ammonium polyphosphate (APP) and disodium octaborate tetrahydrate (DOT)) and two ultraviolet absorbers (UV-531 and nanoTiO_2) for flame retardancy of bamboo were selected and added to waterborne polyurethane (WPU) to make composite coatings with flame retardancy and weathering resistance. The coated sheets were analyzed after flame retardancy and outdoor aging to determine the suitable conditions for flame retardant and anti-aging modification, which provides a reference for the industrial production of bamboo integrated materials.

MATERIAL AND METHODS

Moso bamboo (*Phyllostachys edulis* (Carr.) H.de Lehaie) was taken from Jiangxi Province, China. The bamboo strips were hot-pressed into the laminated bamboo with dimensions of 100 mm (tangential) × 100 mm (longitudinal) × 9 mm (radial). The adhesive was phenolic glue, the hot-pressing temperature was 130℃, the hot-pressing pressure was 3.9-5.9MPa, and the pressure was segmented, from low to high, and the hot-pressing time was 1.5-2.0 h, radial fit between them. The moisture content of bamboo lumber was maintained at 4% to 6%. SETAQUA 6515 water-based paint was purchased from Allnex Resin Co., Ltd. (Shanghai, China), with polyurethane as a film-forming component and the solid content was 50%, purchased from Damao Chemical Reagent Factory (Tianjin, China). Boric acid (BA, AR, purity>99%) and borax (BX, AR, purity>99%) was obtained from Damao Chemical Reagent Factory (Tianjin China). Ammonium polyphosphate (APP, AR, purity>99%) and disodium octaborate tetrahydrate (DOT, AR, purity>99%) were provided by Solomon Biotechnology Co., LTD (Tianjin China). Nano-TiO_2 (5-10nm) purchased from RON Reagent Co., Ltd. (Tianjin China). 2-hydroxy-4-n-octoxy-benzophenone (UV-531) co., LTD (Tianjin China).

Preparation of coating

WPU coatings were modified with four types of bamboo preservatives compounded with two types of UV absorbents as shown in Tab. 1. WPU coating was diluted from 50% solid content to 20% and mixed with one type of these ultraviolet light absorbents at room temperature for 8h, the magnetic stirrer was used for stirring, stirring speed 100 r/min. The added amount of the ultraviolet light absorbent was 1% (mass fraction) of the target coating, and then mixed with one type of these four compound preservatives for 2h. The modified WPU coating was stirred completely before painting.

Tab. 1: Modifications of WPU coatings.

Specimens	Preservatives	Preservative components ratio	UV absorbents
0	WPU	1	-
1	BA/BX/WPU	1:1:8	UV-531
2	BA/BX/APP/WPU	75:75:7:600	
3	BA/BX/DOT/WPU	75:75:42:600	
4	BA/BX/APP/DOT/WPU	75:75:7:42:600	
5	BA/BX/WPU	1:1:8	nano-TiO_2

The bamboo laminate was polished with 200-grit sandpaper to get a smooth surface. The modified WPU coating was applied on the surface of the laminated bamboo with a brush, with a balance for weighing, each brushing 2 g, and then dry in natural conditions for 1 day, and then after reaching the finger dry brushing next time, a total of 4 times. Then the natural air drying for 3 days reserve, 3 samples of each coating combination were repeatedly prepared for performance testing.

Thermogravimetric test method

Thermogravimetric analysis (TGA) was performed on the selected bamboo samples painted with 0, 1, 2, 3, 4, 5 paints and raw material (RM).

Each sample was about 10.00 mg and was analyzed with Q500 (TA Instruments, USA). The temperature is heated at the rate of 20℃/min in the nitrogen atmosphere, and the temperature range of the test is 0℃ ~ 800℃.

Flame retardant test method

Cone calorimetry is one of the important methods to detect the combustion of simulated real fire materials. The principle of heat generated by each 1g oxygen consumed by materials is used to reflect the real combustion of materials. The samples were heated up to 600℃ using a rate of 10℃/min. Flammability of the samples was measured by a cone calorimeter (Fire Testing Technology, UK) according to ISO 5660-1. Samples with the dimensions of 100 mm × 100 mm × 9 mm were wrapped by aluminum foil and then irradiated horizontally under a heat flux of 50 kW/m^2, the test lasts ten minutes, and recorded the residual carbon morphology. The main parameters investigated are: heat release rate (HRR), total heat release (THR), smoke release rate (SPR), smoke release amount (TSP), mass loss rate (MLR), specific extinction area (SEA), average heat release rate (ARHE), etc. The main parameters investigated are: heat release rate (HRR), average heat release rate (ARHE), total heat release (THR), smoke release amount (TSP), mass loss rate (MLR), etc.

Aging resistance test method

In order to be closer to the aging condition of bamboo under natural conditions, the aging resistance test was carried out at a place with an average temperature of 3.9℃, an average precipitation of 0.34mm, a longitude of 117.709404 and a latitude of 39.087068. Bamboo samples treated under different conditions (100mm×100mm×9mm) were placed under outdoor natural conditions to observe the characteristics of discoloration, loss of light, cracking, peeling, powder and other properties of the paint film, and were sampled and evaluated every other month for a total of two months.

RESULTS AND DISCUSSION

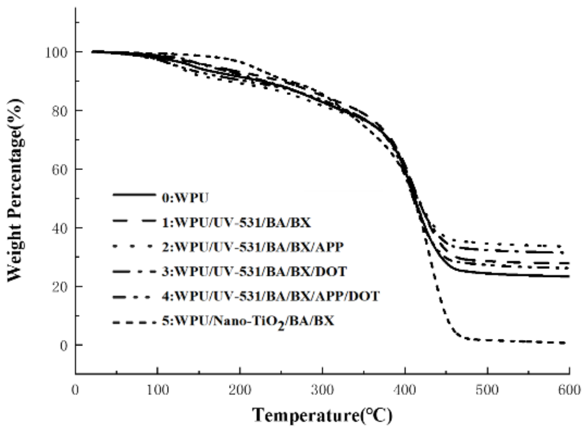
Thermogravimetric analysis (TGA)

TGA was employed to investigate thermal stability and thermal degradation behaviors of test materials treated with different coatings. Fig. 1 and Fig. 2 show the TGA and Derivative Thermogravimetry (DTG) curves under N_2 atmosphere. The decomposition process of WPU with flame retardants and UV absorbers is roughly divided into two stages of decomposition: the first stage is roughly 0-350°C, the hard segment molecular chain and flame-retardant substances are decomposed, mainly in WPU, the $-CH_3$ and -COOH in the medium begin to decompose; the second stage is roughly 350-475℃, the soft segment molecular chain is broken and decomposed, mainly the breaking of the Carbamate and the urea linkage. This is similar to the experimental analysis by Yan et al. (2021) and Ullah et al. (2017). After 475℃, the material is basically decomposed, and the quality does not change.

The addition of flame retardants brought forward the initial decomposition temperature of the coatings. Boron flame retardants began to decompose first, BX and DOT began to decompose at about 50℃, BA began to decompose at about 102℃, and gradually generated metaboric acid, pyroboric acid, Boron trioxide, the final molten glass-like substance, boron trioxide, coats the surface of the bamboo, preventing further combustion of the bamboo, and plays the role of heat insulation and oxygen insulation (Gurwinder et al. 2021, Yu et al. 2017).

APP is an inorganic flame retardant with phosphorus and nitrogen content. It decomposes at about 270□ and gradually generates polyphosphoric acid and ammonia gas. The generated polyphosphoric acid is a strong dehydrating agent to promote the dehydration and carbonization of the bamboo surface, and the non-volatile substances cover the surface of the substrate to further isolate oxygen, and the generated ammonia and water vapor will further slowdown the combustion speed (Zhang et al., 2020). Compared with other coatings, the thermal decomposition temperature of coating 5 with nano-TiO_2 was delayed. It may be that the network-like nano-TiO_2 was mixed with the coating to cover the surface of the substrate, which enhanced the stability of the substrate. And with the synergistic effect of BA and BX flame retardants, it effectively slowed the weight loss rate at the beginning. The peak apex of the DTG curve corresponds to the inflection point of the TG curve, which is the maximum point of the weight loss rate. The number of peaks in the DTG curve is equal to the number of steps in the TG curve. The area of the DTG curve is proportional to the weight loss.

The addition of flame retardants increased the amount of carbon residue in the paint. It can be seen from the figure that the final carbon residue in the paint 2 > 3 > 1 > 4 > 0 > 5, compared to the unmodified coating 0 (20% WPU), the residual carbon content of modified coatings 1, 2, 3, and 4 all increased significantly, and the improvement of coating 2 was the most obvious, and the residual carbon content increased by 59%. Ammonium polyphosphate has a synergistic flame-retardant effect with boric acid and borax, and the expanded carbon layer can further heat insulation and oxygen. This is consistent with the results reported by Hafez and Tajvidi (2020) that the isomerization reaction of newly formed polymer materials under the catalysis of boric acid may promote the formation of carbonization.



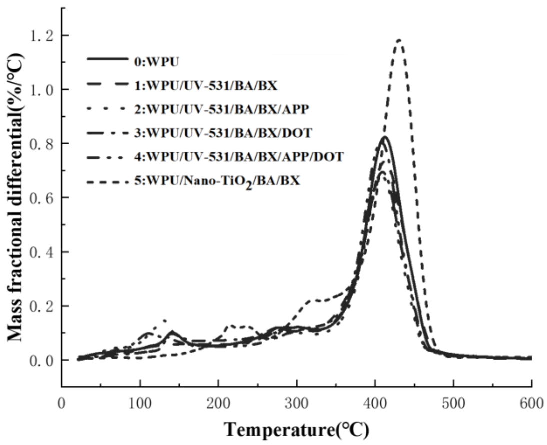


Fig. 1: TGA curve of coating. *Fig. 2: DTG curve of coating.*

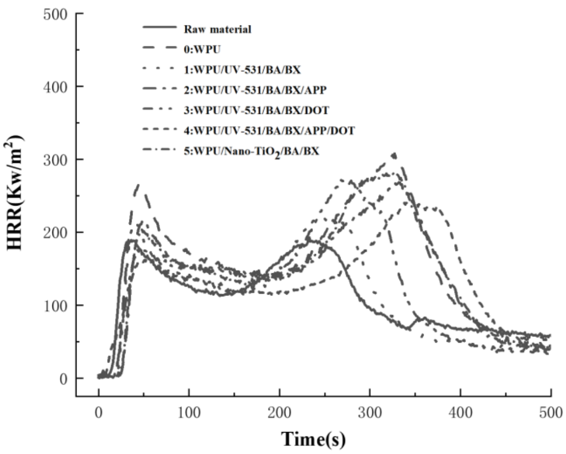
Analysis of flame retardancy

Heat release analysis

The use of cone calorimeter studies the combustion properties of screened test material. five kinds of coatings screened through the analysis of physical and chemical properties of coatings plus control materials, a total of six, in the burning test intense combustion. The larger the heat release rate (HRR) and peak heat release rate (pkHRR) values, the faster the flame propagation during pyrolysis and combustion. It can be seen from Tab. 2 and Fig. 3 that the heat release of the six test materials has a bimodal characteristic curve of bamboo materials.

Tab. 2: Coating pkHRR value.

The serial number	RM	0	1	2	3	4	5
F-pkHRR /(kW·m^2)	189.07±3.58	265.77±6.37	215.56±4.27	215.40±6.31	175.75±5.51	163.17±4.43	203.50±5.76
Time to peak/s	36±2.60	45±3.33	41±3.41	49±4.42	47±3.63	53±2.56	48±3.65
S-pkHRR /(kW·m^2)	192.76±3.49	307.78±4.42	218.44±5.36	268.46±6.44	271.48±5.60	243.63±3.51	281.60±4.82
Time to peak/s	233±5.46	327±3.39	234±5.43	332±3.51	271±6.54	347±5.42	326±3.07



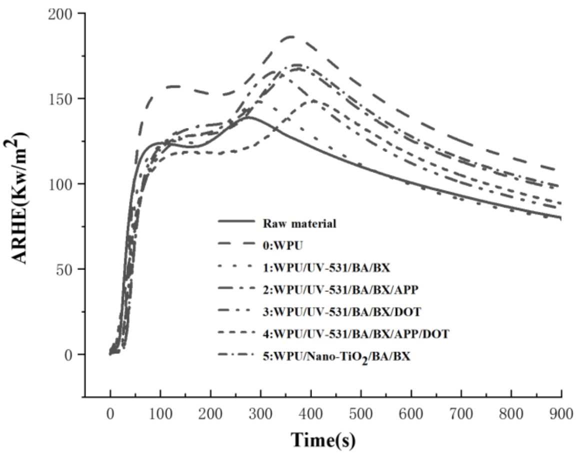


Fig. 3: HRR curve of coating material. *Fig. 4: ARHE curve of coating material.*

The first peak heat release rate(F-pkHRR) appeared at 36-53 s in the first stage. Compared with test material 0, the time was delayed. And the F-pkHRR time of test material 4 was the latest, which appeared at 53 s, delayed by 17 s. Wang et al. (2022) suggested this could be because APP and boron-based flame retardant have synergistic flame retardant, and APP generates phosphoric acid and metaphosphoric acid at high temperature, which has a strong dehydration and carbonization effect. Boron compounds generated molten boron trioxide under high temperature conditions, covering the surface of the substrate, further thermal insulation and oxygen isolation, and delayed the appearance of the peak heat release. From the pkHRR value, test material 3 and test material 4 decreased by 7.05% and 13.70% respectively compared with the material, and the modified coatings 1, 2, 3, 4, and 5 were compared with the test materials 0 with 20% WPU, the pkHRR value decreased. Among them, the reduction of test material 4 is at most 38.60%, which further proves the synergistic flame-retardant effect of boron compounds and APP. This result is consistent with the research conclusion reported by Xu et al. (2018) on BA fireproof coatings.

The second phase had a second heat release peak at 233-347 s. Compared with the material 0, the time when the second heat release peak appeared was also delayed, the second heat release peak time of test material 4 was the latest at 347 s, which was delayed by 114 s. The pkHRR value of the modified sample decreased, and the value of the sample 1 decreased by up to 29.03%. A dense carbon layer was formed during the combustion process, further Delay burning. From the graph of the average heat release rate (ARHE) in Fig. 4, a similar conclusion can be drawn. A second average heat release rate appears nearby, with all modified coatings having a lower average heat release rate than the unmodified sample 0, the heat release peak of the modified coating after adding the flame retardant was lower than that of the unmodified coating, and the peak time was also delayed, and the flame-retardant effect was obvious.

From Fig. 5, the curve in the first stage (0-300 s) is the stage of rapid thermal decomposition of bamboo silk, and the second stage (300-900 s) includes slow thermal decomposition and the formation of carbon layer. Different flame-retardant treatments had a significant effect on its combustion, reducing of heat released. The total heat release of test material 0 (20% WPU) was the largest. Compared with the unpainted material, the heat release of test material 2, 3, 4, and 5 all increased, indicating that the added WPU, flame retardant and UV light absorbers also release some of the heat during combustion. Test material 1 had the lowest heat release, probably because the water vapor produced by BA and BX absorbed part of the heat to a certain extent, finally generated 71.66 MJ/m^2 of combustion heat, which was 26.32% lower than the 97.26% heat generated by the unmodified test material 0, and had a good flame-retardant effect. After 900s, the heat release curve of the specimen tends to be flat, and the combustion process basically ends. Gu et al. (2020) suggested that it's the carbonization layer formed by borates inhibits the diffusion and heat transfer of volatile gases during combustion.

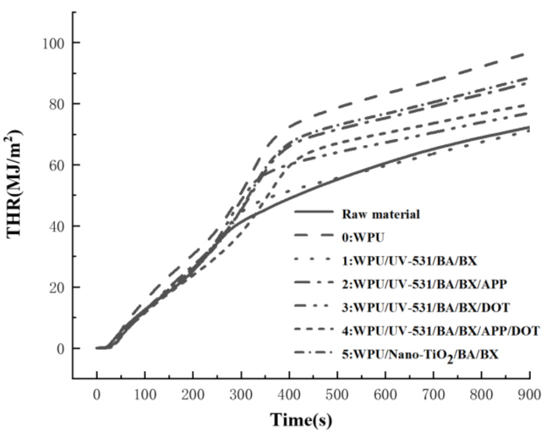


Fig. 5: THR curve of coating material.

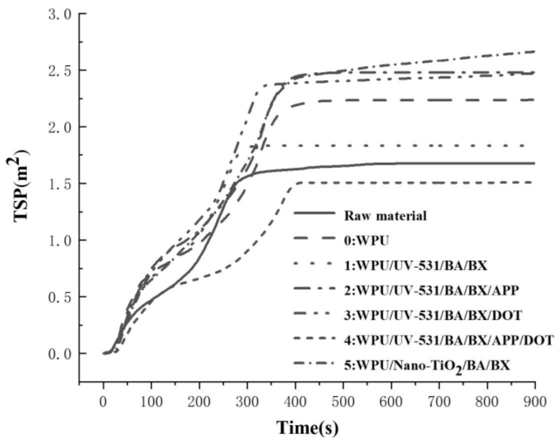


Fig. 6: TSP curve of coating material.

Smoke release analysis

Compared with the materials, Fig. 6 shows that only the material coated with test material 4 (BA/BX/DOT/APP) has a lower smoke emission than the material, with better smoke suppression effect may be due to the synergistic effect of APP and boron compounds, which inhibits the generation of smoke. Hao et al. (2022) modified poplar wood with APP impregnation also successfully obtained low heat release rate and excellent smoke suppression effect. Compared with the unmodified test material 0, the smoke emission of test materials 1 and 4 was reduced, indicating that these two coatings have better smoke suppression. After 400 s, the total amount of flue gas generated basically did not change.

It can be seen from Tab. 3 that the flame retardant based on boron compounds is added to the WPU, which greatly reduces the generation rate of CO and CO_2. The CO generation rate of test materials 2 and 4 is only 10% of the RM, and the CO_2 generation rate is only 4% of the RM. Compared with unmodified sample 0, the production rates of CO and CO_2 in samples 1, 2, 3, 4, and 5 were all lower. It was demonstrated that the addition of boron flame retardants has a positive effect on inhibiting the rate of CO and CO_2 generation, and many studies have also proved the good smoke suppression properties of boron compound flame retardants. Xu et al. (2018) also reached the same conclusion in the study of boric acid as a synergist to improve the flame retardancy and smoke suppression performance of transparent expansive fire-retardant coatings. Xu speculated that this effect can be ascribed to the fact that the residual masses of the coatings noticeably increase with increase loading of BA. Generally, the increase in residual mass is beneficial for reducing the release of combustible gases and smoke precursors, thus resulting in less smoke production and heat release during burning. At the same time, APP and boron compounds are synergistic flame retardant, and metaphosphate generated by APP promotes dehydration and carbonization on the surface of bamboo. In addition, boron substances generate boron trioxide at high temperature and adhere to the surface of the substrate, which inhibits the generation of CO and CO_2.

Tab. 3: Average CO and CO_2 yield.

The serial number	RM	0	1	2	3	4	5
CO average yield/(kg•kg^{-1})	0.07±0.02	0.01±0.01	0.01±0.03	0.001±0.004	0.002±0.001	0.001±0.001	0.002±0.002
CO_2 average yield /(kg•kg^{-1})	1.82±0.43	0.95±0.36	0.31±0.11	0.08±0.03	0.09±0.03	0.07±0.01	0.79±0.21

Mass loss analysis

The residual quality of bamboo is shown in Fig. 7, and the mass loss of the seven samples is roughly divided into two stages. The first stage was 0-400°C. At this stage, the quality of bamboo material declined rapidly, which belonged to the rapid combustion stage of flaming combustion, mainly the dehydration of bamboo material, and the decomposition of hemicellulose, lignin and cellulose. The second stage was after 400°C. this stage was the calcination stage, and the quality of the bamboo material basically stabilized. Under high temperature, the glass-like substance generated by the boron compound covered the surface of the substrate, preventing further volatilization of the substrate components (Gu et al. 2020).

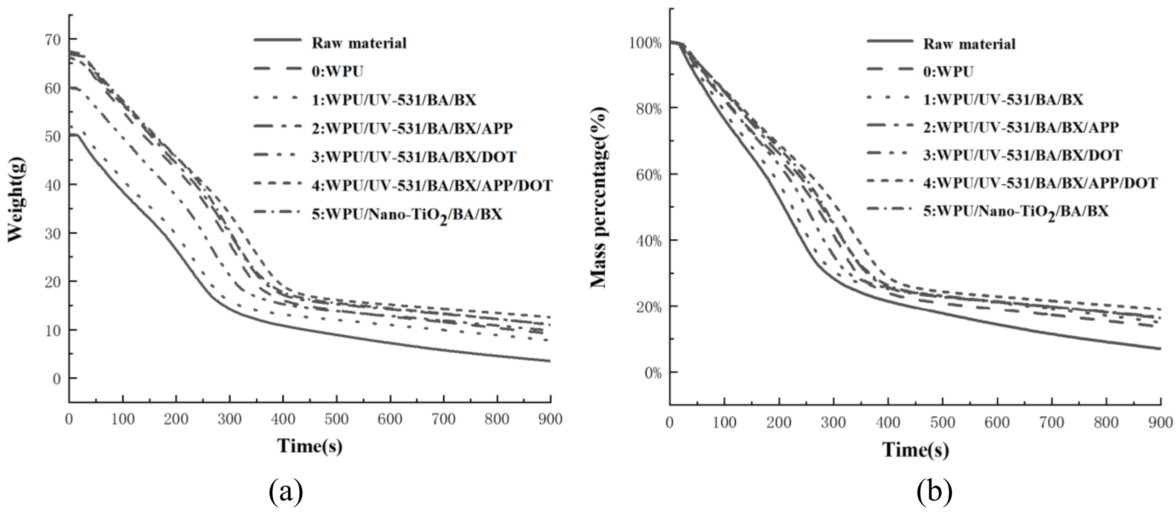


Fig. 7: Curve of coating quality change (a is the weight loss over time, b is the mass percentage loss over time).

At any stage of combustion, the quality change of the coated bamboo was smaller than that of the untreated bamboo, and the final residual carbon quality was also higher than that of the raw material. It can be seen that the coating has an impact on the pyrolysis process of the material. Uner et al. (2016) also experimented and reported the effects of borate on maximum degradation temperature and carbon residue: Higher concentration levels of borates resulted in higher char content of Oriental beech wood specimens. Uddin et al. (2017) have reported experiments that are also consistent with this conclusion. The final carbon residues of test materials 1, 2, 3, 4, and 5 with flame retardants were higher than those of test material 0 (20% WPU), which also reflected the boron and phosphorus added to the coating from the side. Flame retardants will eventually increase the amount of carbon residue, and the formed expanded carbon layer can reduce combustion.

Carbon residue morphology analysis

The comparison of the residual charcoal between the material and the test material coated with different coatings is shown in Fig. 8. It can be seen that the material board is white and gray after burning, and the shape is irregular. The test material 0, which was only coated with water-based polyurethane, was also white and gray bamboo strips after burning, but the shape was relatively regular, while the test materials 1, 2, 3, 4, and 5 with the flame retardant were burned after burning. Black or dark gray color, which proves that the addition of flame retardant increases the amount of carbon residue, which is beneficial to reduce heat release and improve the flame-retardant effect. The study results were consistent with those of He et al. (2019), who reported that borate can effectively improve the flame retardancy of porous pulp foam. He pointed out that coke is an important indicator of material flammability reduction. Coke production increases with the increase of borate load. Similarly, the formed carbon layer can act as a thermal barrier to protect the foam from further degradation. Therefore, borate has a significant effect on the flame-retardant effect. In terms of color, test material 4 appeared carbon black, and the final arrangement was the most regular, indicating that the added BA and BX, APP and DOT played a synergistic flame-retardant effect, and the residual carbon content There is a great improvement, which proves the residual quality from the side and has a better flame-retardant effect.

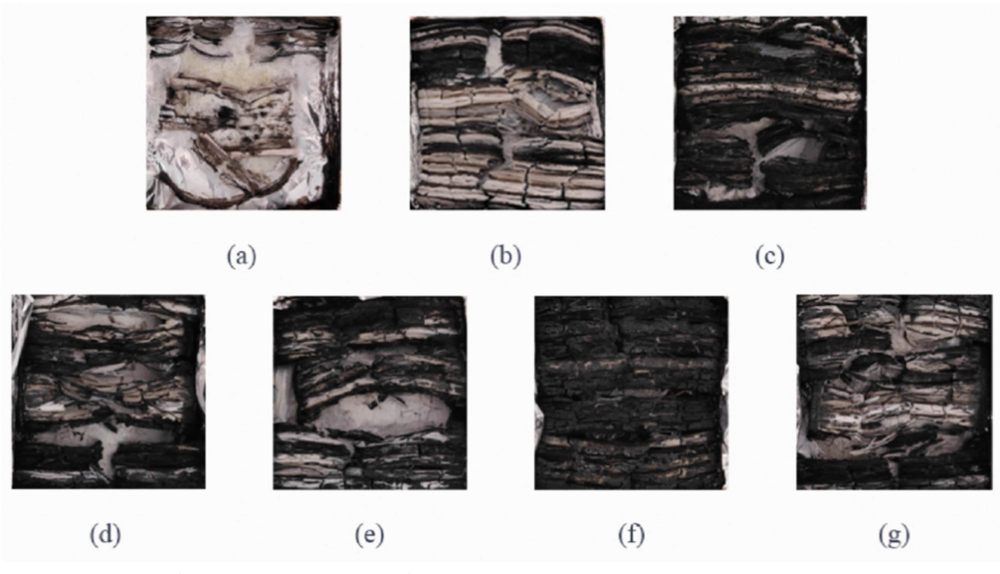


Fig. 8: Morphology of carbon residue of coating: a) raw material, b) 0, c) 1, d) 2, e) 3, f) 4, g) 5.

Anti-aging test analysis

As can be seen from Tab. 4, coatings 1 and 5 did not fall off after outdoor aging, and still have good adhesion. Coatings 2,3,4 were partially shed, and the adhesion decreased, and the adhesion showed a downward trend. After two months, the adhesion of test materials 0 and 1 decreased to grade 2, and the adhesion of test materials 2,3,4,5 decreased to grade 4.

Tab. 4: Aging resistance of coating.

	Untreated		**Treated for a month**		**Treated for two months**			
	Coating shedding	**Adhesive force**	**Coating shedding**	**Adhesive force**	**Adhesive force**	**L***	**a***	**b***
Specimens	-	-	-	-	-	56.53	6.51	23.55
0	-	Level 1	-	Level 1	Level 2	60.59	8.57	27.2
1	-	Level 1	-	Level 1	Level 2	67.62	3.59	12.89
2	-	Level 1	Partial	Level 3	Level 4	70.12	3.21	12.01
3	-	Level 2	Partial	Level 3	Level 4	74.51	2.16	9.09
4	-	Level 2	Partial	Level 4	Level 4	73.45	2.83	11.07
5	-	Level 1	-	Level 2	Level 4	76.13	2.29	7.38

It was found that the surface color parameters of the test materials were collected by the color meter: compared with the material, the test materials ΔL* increased, Δa*, Δb* decreased. After 2 months of outdoor treatment, the brightness of the unmodified test material was significantly lower than that of the modified test material, indicating that visible light and ultraviolet light reduced the brightness of the test material and deepened the red and yellow color change. The light stability of the test material added with UV absorbent is obviously higher than that without UV absorbent 0, indicating that UV-531 and nano-TiO_2 can significantly improve the aging resistance of the test material. Rao et al. (2022) summarized the methods of UV modifiers to improve the photostability of bamboo, and Tang et al. (2022) also reported the effect of nano-TiO_2 coating on the photoaging properties of bamboo.

CONCLUSIONS

Combined with recent environmental protection water-based coatings, the article uses bamboo laminates as the base material, adding 2 kinds (UV-531, nano-TiO_2) ultraviolet light absorbers, 4 kinds (BA/BX, BA/BX/APP, BA/BX/DOT, BA/BX/DOT/APP) compound flame retardant. 6 kinds of modified WPU coatings were prepared by physical mixing, and the modified coatings were applied to the surface of bamboo laminates. TGA and cone calorimetry experiments were performed to analyze the thermal decomposition properties of the finishing materials, and further comparisons were made by aging analysis.

The optimal components and proportions with excellent physical and chemical properties, flame retardant and anti-aging properties were screened out through experiments. The best coating formula is 20% solid content of WPU, 8 g coating amount of WPU/BA/BX/APP/DOT/water/UV-531 modifier, BA/BX/APP/DOT/water quality ratio is 75:75:7:42:600, and the amount of UV-531 added is 1% of the coating mass.

With the shortage of wood resources, the promotion of bamboo has been put on the agenda, bamboo is recognized for its excellent mechanical properties, green environmental protection, short growth cycle and other advantages. We need to invest more resources to the development of bamboo, so that it can be widely used in construction, packaging, and new types of bamboo plastics and other new material fields. Throughout the development of bamboo flame retardant technology, the future should focus on the following aspects of research: *(1)* Improve the compatibility between nanomaterials and coatings to avoid agglomeration and accumulation; *(2)* The development of "one agent multi-functional coatings", in addition to the function of flame retardant and anti-aging properties, increase the bamboo waterproof, mildew resistance, insect resistance and other research; *(3)* Water-based polyurethane coatings should also increase efforts to research, synthesized by chemical means instead of buying.

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