Formulation of Flame-Retardant Waxes and Evaluation of Combustion Characteristics of Treated Wood Using Mass Loss Calorimetry

Hyung Woo Lee,[a] Jae Hee Jung,[a] Jong-Shin Lee,[b] Soon Park,[c]
and Seon-Mee Yoon [c,*]

The objective of this study was to synthesize flame-retardant wax by nano-sizing a previously developed flame-retardant with favorable performance characteristics. Additionally, this study assessed the leaching resistance of wood treated with flame-retardant wax. The findings revealed that wood treated with flame-retardant wax demonstrated no notable change in the weight loss rate before and after weathering treatments. In contrast, wood treated exclusively with flame retardants exhibited a discernible increase in the extent of weight loss. Furthermore, the leached phosphorus content was measured during the weathering process, revealing that the amount extracted from wood treated with flame-retardant wax was approximately 1/20 times of that extracted from wood treated with flame-retardant agents. Therefore, that wood treated with flame-retardant wax demonstrated superior flame-retardant performance before and after weathering treatments.

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*Contact information: a: Wood Engineering Division, Forest Products and Industry Department, National Institute of Forest Science, 57 Hoegi-ro, Dongdaemun-gu, Seoul 02455, Korea; b: Professor, Department of Biobased Materials, Chungnam National University, Deajon 34134, Korea; c: Lumber One Labs Co., Ltd, Deajeon 34134, Korea; * Corresponding author: bysim2215@gmail.com*

INTRODUCTION

The incidence of natural disasters has been increasing globally, with extreme weather events becoming increasingly prevalent due to climate change. Climate change is largely attributed to global warming, which can be primarily attributed to the rise in atmospheric concentrations of greenhouse gases (GHGs), such as carbon dioxide (CO_2), due to the extensive use of fossil fuels (Bilgen *et al.* 2008).

Forests serve as primary absorbers of carbon dioxide, which is a primary driver of global warming. The wood produced by the trees in these forests serves as a significant reservoir of carbon dioxide. The accumulation of wood, which acts as a carbon dioxide reservoir, has the potential to increase the amount of bound carbon, thereby contributing significantly to the mitigation of climate change and global warming.

Wood serves a dual function as both a carbon reservoir and as a renewable resource, in addition to being an environmentally friendly building material (Ramage *et al*. 2017; Maier 2021). A considerable quantity of CO_2 is emitted into the atmosphere throughout the lifecycle of a building. This encompasses the production of building materials (BM) and construction of the building itself (Joseph and Tretsiakova-McNally 2010). In the case of buildings constructed with wood, the generation of GHGs during the construction process can be reduced compared to buildings constructed with concrete. Börjesson *et al*. (1999) determined that the primary energy input (primarily derived from fossil fuels) in the production of building materials was approximately 60% to 80% higher when concrete frames were considered in comparison to wood frames. Additionally, enhancing the thermal insulation of a building can minimize the heat loss generated and reduce the energy used in the building. Moreover, wood manufacturing requires less energy than alternative materials. The carbon dioxide emissions associated with the production of wood are estimated to be approximately 0.5% of those from steel and 0.13% of those from aluminum. Consequently, there is a growing trend toward the use of wood in building structures on a global scale.

Wood is a highly advantageous material for construction; however, it also presents a significant disadvantage, as it is highly susceptible to combustion. To facilitate the large-scale and high-rise development of wooden constructions, it is of utmost importance to ensure fire safety. In South Korea, buildings comprising five or more stories are required to comply with a two-hour fire resistance standard for their structural components. In addition, Korea has established a classification system for building finish materials based on their fire-resistance performance, categorizing them as noncombustible, quasi-noncombustible, or flame-retardant. This system mandates the use of these materials according to their designated classifications (Ministry of Land, Infrastructure and Transport 2018).

The use of flame-retardant-treated wood as an exterior material in timber construction is a potential issue, particularly when exposed to these elements. Prolonged exposure to precipitation and environmental conditions has raised concerns regarding the leaching of flame retardant chemicals. This phenomenon can affect the efficacy of the flame-retardant treatment, thereby influencing the overall durability and performance of the material when used in exterior applications (Baysal *et al*. 2006; Harada *et al*. 2009).

To prevent the wood deterioration caused by microorganisms, it is essential to assess the resistance of wood preservatives to moisture leaching. Leaching of wood preservatives is typically associated with a reduction in the lifespan of wood and adverse effects on the environment and human health (Barbero-López *et al*. 2021). The regulatory standards for wood preservatives across all countries worldwide stipulate that the preservative must be fixed within the treated wood, with a particular focus on its resistance to leaching (EN 351 2012; CSA O80 2014; AWPA U1-18 2024). Currently, there is no flame-retardant agent for wood that is resistant to moisture leaching. Consequently, no agent that is effectively fixed and rendered insoluble within the treated wood structure has yet been developed. No country has established regulations or standards to address this issue. The creation of fixative flame-retardant agents for wood is crucial for guaranteeing the fire safety of wooden structures and facilitating their wider utilization.

Recently, technological solutions have been developed and introduced in South Korea with the objective of enhancing the leaching resistance of wood flame retardants against moisture. These techniques include mixing flame retardants with water-soluble resins (Park *et al*. 2023) and double impregnation treatments, in which a flame retardant is first applied, followed by treatment with hydrophobic wax (Jung and Lee 2023). The application of wax to wood surfaces has been shown to enhance dimensional stability in the face of expose to moisture, whilst concurrently mitigating the degradation of flame-retardant properties caused by external factors. Nevertheless, concerns have been raised regarding the generation of toxic gases during the combustion of flame-retardant wood treated with synthetic resins. Furthermore, double impregnation treatments, which entail the production of flame-retardant wood through two distinct impregnation processes, have resulted in challenges such as diminished on-site workability, reduced productivity, and elevated production costs.

The objective of this study was to manufacture a flame-retardant wax by nano-sizing a previously developed and recognized flame-retardant material that demonstrated excellent performance and uniform dispersion in wax. The leaching resistance of the wood treated with this flame-retardant wax was then evaluated.

EXPERIMENTAL

Materials

Wood

To achieve a uniform impregnation of the flame-retardant, diffuse-porous maple wood (*Acer saccharum*) was utilized, with a specimen measuring 95 mm (length) × 95 mm (radial) × 25 mm (tangential). The moisture content of the air-dried specimen was 9.6%. The number of replicates for each specimen type was five.

Flame retardant

The flame retardant utilized was minalith, which complies with the Type 3 standard set forth by the American Wood Protection Association (AWPA U1-18 2024). Minalith is a white powder composed of ammonium sulfate ($(NH_4)_2SO_4$), boric acid (H_3BO_3), diammonium phosphate ($(NH_4)_2HPO_4$), and sodium borate ($Na_2B_4O_7$). The composition of minalith is presented in Table 1.

Table 1. Chemical Composition of Minalith (Unit: %)

Chemicals	Composition
Ammonium sulfate ($(NH_4)_2SO_4$),	60.0
Boric acid (H_3BO_3)	20.0
Diammonium phosphate ($(NH_4)_2HPO_4$),	10.0
Sodium borate ($Na_2B_4O_7$).	10.0

Fisher-Tropsch wax

To achieve uniform dispersion of the flame-retardant agent, Fischer-Tropsch wax (FT-wax), which is harmless to humans, was employed. FT wax is byproduct wax generated in the production of liquid fuels and other products from coal and natural gas. It is synthesized from carbon monoxide and hydrogen as shown Eq. 1.

$$(2n + 1)H_2 + nCO \rightarrow C_nH_{2n+2} + nH_2O \qquad (1)$$

FT Wax exhibits a high degree of crystallinity, a linear molecular structure, high thermal stability at elevated temperatures (high melting points), low molecular weight, and high hardness. It is employed in various fields, including polyvinyl chloride (PVC), hot-melt adhesives (HMA), and interpolymer complexes (IPC). A detailed account of the properties of FT Wax is provided in Table 2.

Table 2. Properties of Fischer-Tropsch Wax

Typical Properties	FT Wax
Melting point (°C)	80-115
Viscosity at 135 °C centipoise	>10
Molecular Structure	Linear
Molecular Weight: Daltons	600-1300

Test Methods

Flame-retardant wax formulation

To achieve uniform dispersion of the flame retardant in FT-Wax, the two substances were placed in a ball mill and subjected to attrition milling (ATTRITION MILL-KMDC-18). This yielded flame-retardant wax with nano-sized particles.

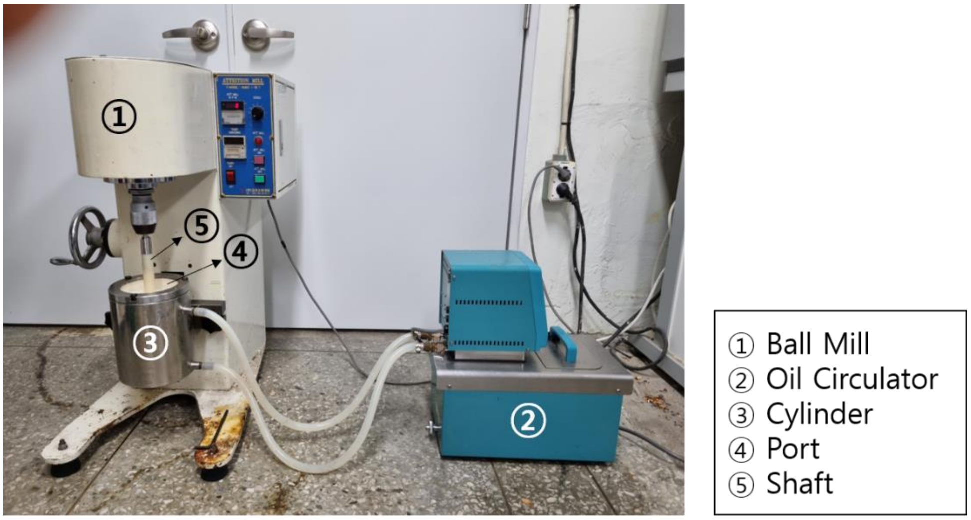
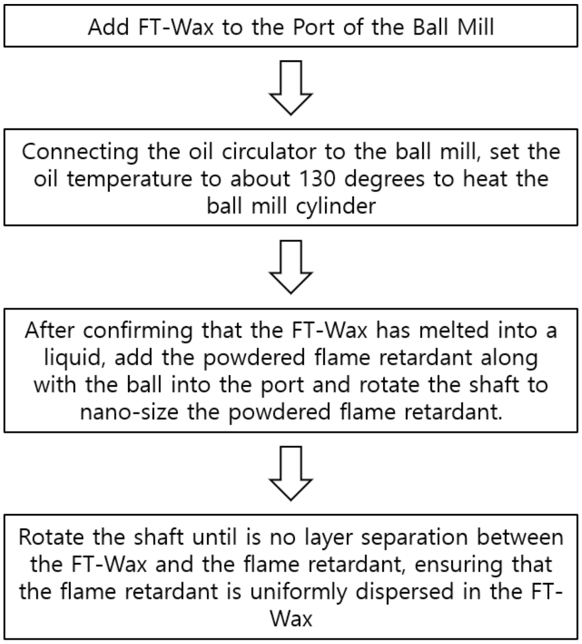


Fig. 1. Schematic of ball mill machine



Add FT-Wax to the Port of the Ball Mill

Connecting the oil circulator to the ball mill, set the oil temperature to about 130 degrees to heat the ball mill cylinder

After confirming that the FT-Wax has melted into a liquid, add the powdered flame retardant along with the ball into the port and rotate the shaft to nano-size the powdered flame retardant.

Rotate the shaft until is no layer separation between the FT-Wax and the flame retardant, ensuring that the flame retardant is uniformly dispersed in the FT-Wax

Fig. 2. Preparation procedure for flame-retardant wax

During the nano-sizing treatment, samples of the FT-Wax solution containing the flame-retardant were periodically collected and heated to a high temperature. The occurrence of layer separation between the flame retardant and FT-Wax was monitored, and ball milling was continued until no layer separation was observed. Although particle size analyzers are available for measuring the size of particles in a liquid, these instruments are only capable of measuring particles dispersed in a liquid at room temperature. Consequently, it was not possible to measure the particle size of the wax solutions at elevated temperatures. Consequently, nano-sizing of the flame retardant was evaluated indirectly by observing the presence or absence of particle precipitation. The methodology employed for the preparation of the flame-retardant wax is shown in Fig. 2.

Impregnation of minalith and flame-retardant wax

To identify the optimal proportion of minalith, flame-retardant solutions were prepared at concentrations of 5%, 10%, and 20%. The specimens were fully immersed in a self-constructed nitrogen gas-pressurized impregnation apparatus, which was then sealed. The pressure impregnation equipment was impregnated with nitrogen gas to increase the pressure to 1.5 MPa, and then the flame-retardant solution was impregnated into the specimen under pressure. In the event of an impregnation of flame-retardant wax, the temperature of the pressurized impregnation device was increased to approximately 120 °C to facilitate the pressurized impregnation. Subsequently, the minalith pressurized impregnation method was employed. The pressure within the device was maintained at approximately 1.5 MPa for 2 h.

Weathering treatment test

Leaching tests were conducted in accordance with KS M 1701 (2018) to investigate the leaching resistance of the specimens treated with minalith alone and those treated with a flame retardant. The specimens treated with minalith alone and those treated with flame-retardant wax were fully immersed in distilled water at a volume 10 times that of the specimens. Subsequently, the mixture was agitated using a magnetic stirrer at 400 to 600 rpm at 25 ± 3 ℃ for 8 h to facilitate the leaching of the water-soluble components of the flame-retardant. Following the leaching process, the surfaces of the specimens were lightly wiped and subsequently dried in an oven set at 60 ± 2 ℃ for 16 h, during which time the volatile components were allowed to evaporate. The aforementioned leaching and evaporation processes were repeated for a total of 10 cycles, with each cycle consisting of the alternation of the two aforementioned steps.

Leaching amount of flame retardant.

Following each cycle of the weathering process, the leachate was collected, and the phosphorus content, which represents the active component of the flame retardant, was measured using a spectrophotometer (DR 3900; Hach USA). Each collected leachate sample was diluted with distilled water at a 1:1000 ratio for phosphorus measurements, and this process was repeated three times to ensure accuracy.

Performance of flame-retardant

The flame retardancy of the samples treated with minalith alone was evaluated through a series of tests including flame-retardant wax before and after weathering, combustion performance tests, and mass loss measurements. The tests were conducted according to the KS F IOS 17445 standard (2013). Prior to testing, the specimens were subjected to conditions of 23 ± 2 °C and 50 ± 5% relative humidity in accordance with ISO 554 (1976) until they reached a constant weight. A mass-loss calorimeter (Festec Co., Korea), which employs a conical radiant electric heater, was used, as illustrated in Fig. 3. The temperatures and measurement times of the calorimeters are listed in Table 3.

Table 3. Conditions of Temperature and Time for Combustion Test

Heater Temperature	Heater Flux Meter	Measure Time
650 to 750 ± 2 °C	5050 ± 0.5 kW/m^2	300 sec

Radiant heat and combustion time were maintained at a constant radiant intensity of 5 at the center of the specimen surface in accordance with KS F 3124 (2022) and KS F IOS 17554 (2013). The combustion tests were conducted approximately 300 s after ignition. The mass loss rate with respect to the combustion time was calculated as a percentage using the initial mass and mass after the 300 seconds combustion test, as illustrated in Eq. 2,

$$\text{Mass loss rate (g/s)} = \frac{m_A - m_B}{t_B - t_A} \tag{2}$$

where m_A is the mass at time A(g), m_B is the mass at time B(g), t_B is the mass at time B(s), and t_A is the mass at time A (s).

The ignition time was determined when the specimen was exposed to radiant heat and generated a sufficient amount of flammable vapor, or when the specimen was ignited by the flame of the igniter installed in the mass loss calorimeter. To examine the charring characteristics of the specimens during combustion following weathering treatments with minalith alone and the flame retardant, specimens that had completed the flame-retardant performance evaluation were collected.

RESULTS AND DISCUSSION

Investigation of Flame-Retardant Performance of Treated Wood According to Minalith Concentration

The objective of this study was to determine the optimal minalith concentration. Table 4 presents the retention of wood treated with minalith at concentrations of 5%, 10%, and 20%. Table 5 shows the flammability of wood treated at each concentration. The amount retained increased in proportion to the concentration of the treated wood. The data indicated that the ignition time exhibited an increasing trend as the minalith concentration increased. In particular, an incremental increase was observed from 0% to 10%, but when the concentration reached 20%, the ignition time exhibited an exponential rise. At each concentration, minalith fully penetrated the cell lumens of the tissues, including the vessels and fibers of the maple wood (Lee *et al*. 2021; Park *et al*. 2023). It is noteworthy that at a minimum concentration of at least 20%, the flame-retardant interacted with the wood to form an effective protective barrier that minimizes the contact between the wood and air during combustion. Furthermore, this interaction markedly inhibited the chemical reactions that occur during combustion.

Table 4. Retention of Wood Impregnated with Mixed Fire-Retardant Solution by Concentration

Concentrations (%)	Retention (kg/m^3)
5	580.7 ± 21.2
10	629.0 ± 18.6
20	647.1 ± 21.7

Table 5. The Conditions of Temperature and Time for Combustion Test

Concentrations (%)	Ignition time (sec)	Weight loss (%)
0	12.5±2.1	24.42±1.27 $^{(a)}$
5	11.0±1.0	18.10±1.15 $^{(b)}$
10	16.7±13.4	18.37±0.25 $^{(b)}$
20	91.7±10.2	15.80±0.50 $^{(c)}$

Note: (a),(b), and (c) indicate statistically significant differences according to Duncan's test (p<0.05).

The weight loss value of specimens treated with 0% minalith was approximately 24.4%, which was approximately 1.34 times higher than that of specimens treated with 5% Minalith. Conversely, an increase in the concentration of minalith from 5% to 10% resulted in a minimal change in the weight loss. The results of the statistical analysis indicated a statistically significant difference between the specimens treated with 0% and 5% minalith ($t^*=0.0031$, $p=0.05$). However, no significant differences were observed between those treated with 5% and 10% ($t^*=0.733$, $p=0.05$). Furthermore, specimens treated with minalith concentrations ranging from 10% to 20% exhibited a significantly lower weight loss, with a reduction of approximately 0.86 times compared to the control group. An investigation of ignition time and weight loss of treated wood at varying concentrations of minalith indicates that a minimum concentration of 20% minalith is required.

Fig. 3. Dispersion state of flame-retardant in wax solution according to ball milling processing time (① prior to ball milling, ② 24 h post-ball milling ③ 36 h post-ball milling)

Investigation of Flame-Retardant Performance

Formation of flame-retardant

The objective of this investigation was to ascertain the extent of homogeneous dispersion of the flame-retardant (minalith) in FT-wax. To this end, flame-retardant samples were prepared in solution at different time intervals, and phase separation was observed between the flame retardant and FT-wax. The results are shown in Fig. 3. Samples 1, 2, and 3 illustrate the retardant solutions prior to ball milling, 24 h post-ball milling, and 36 h post-ball milling, respectively, following the addition of the flame retardant. The precipitation of the flame retardant was observed in samples 1 and 2, whereas no precipitation was observed in sample 3. These findings indicate that ball-milling for a minimum of 36 h is essential for preparing flame-retardant waxes.

Investigation of flame-retardancy performance based on weathering condition

To assess the flame retardancy performance under weathering conditions, the mass loss over the combustion time was investigated, and the results are presented in Table 6 and Fig. 4. The specimens treated with the flame-retardant exhibited an ignition time of approximately 91.7 seconds, while the specimens treated with the flame-retardant had an ignition time of 21.5 seconds, indicating a faster ignition time compared to the specimens treated with the flame-retardant. This can be interpreted as the flame retardant wax coating the surface of the wood, with the ignition point of the coating being lower than that of the wood itself, resulting in a shorter ignition time for specimens treated with the flame retardant wax. A comparison of the ignition times under weathering conditions revealed that the ignition time for specimens treated solely with a flame-retardant was 91.7 seconds prior to weathering and decreased to 10.7 seconds following weathering, indicating a difference of approximately 8.7 times. Furthermore, the ignition times after weathering were shorter than those of the untreated specimens (12.5 seconds. These results indicate that the effective components of the flame retardant exhibited a lack of resistance to leaching.

Table 6. Ignition Time and Mass Loss Rate

Treatments	Ignition time (sec)	Mass loss rate (%)
Non-treatment	12.5±2.12	23.56±1.06 (a)
20% flame-retardant (Minalith) only before weathering	91.7±10.21	15.83±0.55 (b)
20% flame-retardant (Minalith) only after weathering	10.5±2.12	19.53±1.36 (c)
Flame-retardant wax before weathering	21.5±0.71	15.76±0.71 (d)
Flame-retardant wax after weathering	25.5±0.71	15.26±1.61 (d)

Note: (a), (b), (c), and (d) indicate statistically significant differences according to Duncan's test ($p<0.05$).

The mean mass loss for specimens treated exclusively with the flame-retardant was approximately 15.8%, whereas specimens treated with flame-retardant wax exhibited a weight loss of 15.3%. Although the weight losses of both treatments did not demonstrate a statistically significant difference, it was observed that the weight loss for the specimens treated with only the flame-retardant increased to 19.5% following the weathering treatment. This suggests that the flame-retardant components incorporated into the wood may have been dissolved by moisture, resulting in leaching and a subsequent reduction in the flame-retardant efficacy. In contrast, the weight losses for specimens treated with flame-retardant wax were 15.8% and 15.3%, respectively, before and after weathering, demonstrating no significant variation. The results of the Duncan test (t*=0.649; p>0.05) further corroborated the absence of a significant difference, indicating that the effective components of the flame retardant wax injected into the wood were not susceptible to leaching due to moisture.

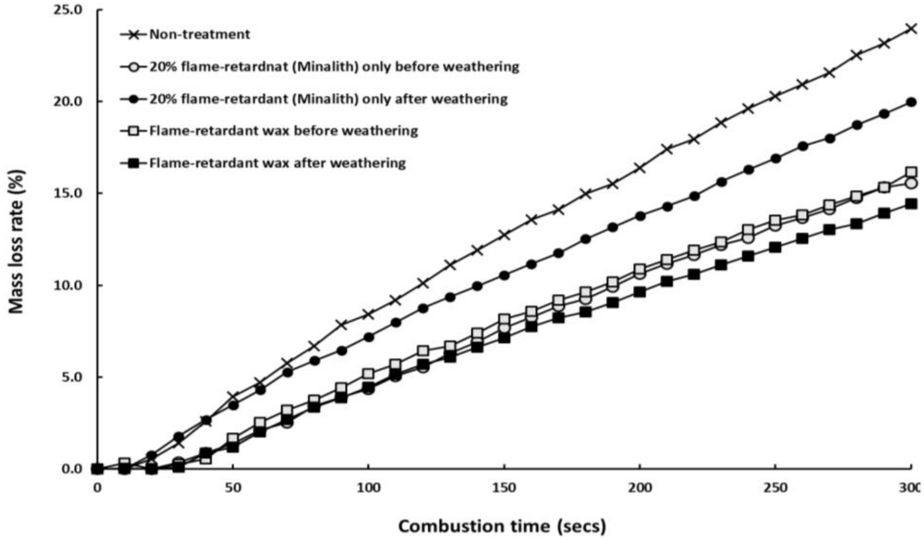


Fig. 4. Mass loss rate by treatment conditions over time during combustion test

Leaching amount of flame-retardant active ingredients according to environmental conditions

The objective of this study was to investigate the leaching amount of phosphorus (P), a principal component of the flame retardant, to ascertain the extent of its leaching potential. The total amount of leached phosphors in the leachate collected after weathering was quantified and is presented in Table 7. The concentration of phosphorus detected in the leachate from specimens treated with only the flame retardant was approximately 0.48 mg/L, whereas that in specimens treated with flame-retardant wax was 0.027 mg/L. This result indicates that the phosphorus leaching amount in specimens treated with flame-retardant wax was approximately one-twentieth that in specimens treated with the flame retardant alone. Thus, it can be concluded that the flame retardant treatment method demonstrated enhanced resistance to leaching due to water compared to the method using the flame retardant alone, indicating an improved flame retardant performance.

Table 7. Amount of Phosphorus Leached from Each Treated Specimen by Weathering Operation

Classification	Phosphors Leaching Amount (mg/L)
Mixed flame-retardant solution	0.48
Flame-retardant wax	0.027

CONCLUSIONS

1. The objective of this study was to determine the optimal concentration of minalith for wood treatment. To this end, the effects of minalith concentrations of 5%, 10%, and 20% on the treated wood were examined. Wood treated with a 20% concentration of minalith exhibited the most effective flame-retardant properties.
2. Following the formation of the flame-retardant wax, sedimentation was no longer a factor after 36 h of ball-milling; therefore, it can be concluded that a minimum ball-milling time of 36 h is required.
3. The investigation of flame-retardant performance in wood treated with flame-retardant wax and flame retardants revealed that there was no significant difference in the weight loss values after weathering between wood treated with flame-retardant wax and other types. Based on these findings, the flame-retardant wax exhibited moisture leaching resistance while maintaining its flame-retardant performance.
4. Investigation of the leaching amount of phosphorus from the flame retardant revealed that the amount of leached phosphorus in wood treated with flame-retardant wax was approximately 1/20 times that found in wood treated with the flame retardant alone. These findings suggest that flame-retardant waxes exhibit markedly enhanced resistance to moisture-induced leaching, coupled with superior flame-retardant efficacy.

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