Effect of Pressure Impregnation with a Boron-Phenolic Composite Flame Retardant on the Combustion Performance and Mechanical Properties of Plywood

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Effects of a boron-phenol-based flame retardant were evaluated relative to the combustion performance and mechanical properties of structural plywood manufactured from two domestic softwood species: larch (*Larix kaempferi*) and Korean pine (*Pinus densiflora*). The flame retardant was applied using a standardized vacuum-pressure impregnation process, and the retention level, combustion resistance, and structural integrity of the treated specimens were determined. The results showed that the treated specimens met the Korean standard (KS F 3113) requirements for bending strength, modulus of elasticity, and water-resistant tensile-shear strength. Larch plywood exhibited modest changes in combustion and mechanical performance, whereas Korean pine showed significant improvements in flame retardancy, including longer ignition time, lower peak heat release rate, and reduced char length and area owing to the higher flame-retardant retention achieved with Korean pine. Furthermore, flame retardant impregnation did not affect adhesive bonding in either species. This study demonstrates that boron–phenol-based flame retardants can effectively enhance fire resistance in structural plywood without compromising its mechanical performance, thus supporting their applicability in manufacturing flame-retardant wood-based construction materials.

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INTRODUCTION

With the increasing use of wood-based materials in architectural and industrial structures, ensuring the flame retardancy of such materials has emerged as a critical issue for minimizing casualties and property damage caused by fire. Plywood products are widely used as a structural and interior material. However, owing to their high flammability and rapid flame spread at elevated temperatures, improvements in fire safety are essential. Methods for enhancing the flame resistance of wood include chemical impregnation, the incorporation of flame retardants into adhesive systems, and surface treatments such as flame-retardant coatings (Grexa *et al.* 1999). Among these, the pressure impregnation method has been recognized as suitable for structural applications because of its ability to deeply penetrate the flame retardants into the wood matrix, resulting in long-term effectiveness.

Among the commonly used flame retardants, boron-based compounds have been applied since the early 20th century to enhance the fire resistance of polymeric materials (Abdalla *et al.* 2003). Representative boron compounds include boric acid (BA) and borax (BX). LeVan and Tran (1990) reported that BA effectively inhibits flame spread but increases smoke production, whereas BX suppresses smoke generation but offers limited flame retardancy. The synergistic combination of BA and BX showed promising potential. These compounds have also been used as wood preservatives and, when exposed to high temperatures, are known to form a glassy film that acts as a physical barrier and inhibits the flow of combustible volatiles to the fire-exposed surface (LeVan and Tran 1990). However, when applied at high concentrations as standalone agents, BA and BX can lead to a reduction in the bending strength and adhesive bonding performance of the treated wood, and they exhibit limitations in fixation stability (Nagieb *et al.* 2011).

In response to these challenges, recent research has focused on resin-based composite systems in which boron compounds are combined with various resin matrices to enhance both the flame retardancy and mechanical performance of wood materials (Dogan *et al.* 2021). Phenol–formaldehyde (PF) resin is widely used in wood modification due to its excellent thermal resistance, weather durability, and inherent flame retardancy (Huang *et al.* 2013). Active research has been conducted on boron-modified phenolic resins (BPF), where boron is chemically introduced into the PF resin structure. The formation of B–O bonds in BPF has been reported to improve thermal stability and increase char yield (Wang *et al.* 2014; Zhang *et al.* 2023). B_2O_3 generated during the combustion of BPF can adhere to the surface of the burning material to form a dense barrier layer, it contributes to self-extinguishing behavior by insulating the underlying material (Abdalla *et al.* 2003). Gao *et al.* (1999) reported that boron-containing PF resins significantly improved the thermal stability and bonding strength of plywood. Bian *et al.* (2015) demonstrated that the incorporation of boron into the PF resin matrix enhanced char residue and resistance to thermal degradation. In addition, Wang *et al.* (2017) found that a combined treatment of wood using low-molecular-weight phenol–melamine–urea–formaldehyde (PMUF) resin and boron compounds markedly reduced peak heat release rate (PHRR) and total heat release (THR), thereby improving the fire stability of the treated wood. Thus, combining PF resins with boron-based flame retardants is considered effective in enhancing fire performance through complementary mechanisms.

Nevertheless, conventional BPF systems are limited by the reactivity of boron during synthesis, increased viscosity, and challenges in process control, which restrict the practically applicable boron concentration (Zhang *et al.* 2023). Moreover, studies evaluating the fire and mechanical performance of high-concentration boron systems applied to wood-based materials remain insufficient.

In this study, a water-soluble PF resin with a low molar ratio was synthesized to enhance reactivity and then combined with a boron compound solution (BAX) containing approximately 45% solids content to develop a boron–phenolic composite flame retardant system (PBAX). This system was applied to structural plywood using a full-cell pressure impregnation process. For the experimental evaluation, larch (*Larix kaempferi*) and Korean pine (*Pinus densiflora*), two species widely used in the Korean structural wood industry, were selected. These species are commonly used in plywood and structural components and exhibit distinct characteristics in terms of chemical treatability. Larch, classified as a refractory species, has dense tissue and abundant resin content, making chemical penetration difficult. In contrast, pine has an open cellular structure that allows for easier chemical impregnation under the same treatment conditions. Therefore, this study aimed to compare the treatment efficiency, fire performance, and mechanical properties of the PBAX system between the two species, and to assess its general applicability and species-specific responses for potential industrial-scale application in flame retardant structural plywood manufacturing.

EXPERIMENTAL

Plywood

Commercial softwood plywood was used as the base material. Plywood specimens were made from larch (*Larix kaempferi*) provided by HUIN Co., Ltd. (Shinan, Korea) and Korean pine (*Pinus densiflora*) supplied by EAGON Industrial Co., Ltd. (Incheon, Korea). The specifications of the test specimens are listed in Table 1. Before testing, all plywood was cut and machined to the required dimensions under controlled environmental conditions, maintaining a moisture content of 8 to 10%. The prepared specimens were then used in a flame-retardant impregnation process.

Table 1. Specifications of Plywood Specimens Used in This Study

Sample	Cross-section Image	Adhesive Type	Thickness (mm)	Composition (ply-veneer thickness)	Application
Larch		PF (Phenol-Formaldehyde)	24	9-ply × 2.2 mm	Structural plywood, Scaffolding
Pine		MUF (Melamine-Urea-Formaldehyde)	15	7-ply × 2.2 mm	Backing board

Flame Retardant

The flame retardant used in this study was prepared by first formulating a boron-based compound (BAX) by mixing boric acid (BA, 99.5% purity) and borax (BX, 99.0% purity) under maximum solubility conditions.

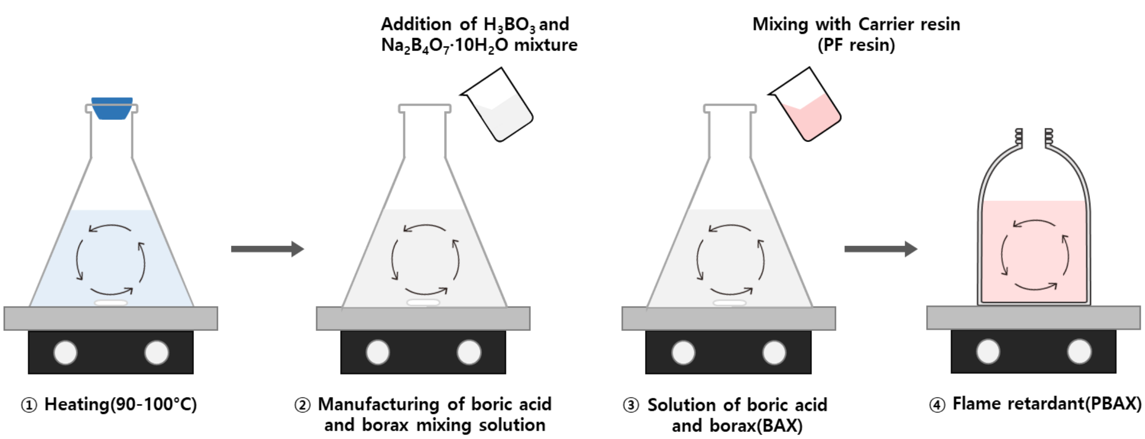

Fig. 1. Flame retardant manufacturing process

The BAX solution had a solid content of 40%. A water-soluble, resole-type phenol formaldehyde (PF) resin with a low molar ratio of 1:1.6 and a solid content of 48.4% was synthesized separately. These two components were then combined in a 1:1 weight ratio (w/w) to produce a boron-phenolic composite flame retardant, referred to as PBAX. The final product was an aqueous resin solution with a solid content of approximately 45%, a pH of 7.38, and a viscosity of less than 100 cP. This PBAX solution served as an injectable agent designed for deep impregnation into the plywood matrix. A schematic of the preparation process is shown in Fig. 1.

Flame Retardant Vacuum Pressure Impregnation Process

The flame retardant plywood was manufactured using the full-cell (Bethell) vacuum pressure impregnation method. The process consisted of three stages: pre-vacuum, pressure, and post-vacuum, as outlined in Table 2. The treatment facilitated deep penetration of the flame retardant into the wood matrix and promoted internal fixation. Following impregnation, the specimens were air dried at room temperature.

The retention level of the flame retardant was calculated using Eq. 1 based on the mass and volume of the specimens before and after impregnation,

$$R = \frac{m_2 - m_1}{V} \tag{1}$$

where R is the retention level (kg/m^3), m_2 is the mass of the specimen after flame retardant impregnation (kg), m_1 is the mass before impregnation (kg), and V is the initial volume of the specimen (m^3).

Table 2. Conditions of Flame Retardant Impregnation Process

Stage	Process Description	Pressure (MPa)	Time (min)
Pre-vacuum	Air evacuation from wood lumens	- 0.08	2
Pressure	Fire retardant impregnation under pressure	1.5	30
Post-vacuum	Excess solution removal and fixation	- 0.08	10

Combustion Performance Evaluation

The flammability of the plywood specimens was evaluated using a 45° flammability tester (FESTEC INTERNATIONAL Co., Ltd., Seoul, Korea). The flame length was fixed at 65 mm and the flame tip was applied to the center of the bottom edge of each specimen for 2 min. In accordance with KS F 2819 (2016), the afterflame and afterglow times were measured following flame exposure. After the soot surface was removed, the char length and area were measured.

Fire performance was further assessed using a cone calorimeter test in accordance with ISO 5660-1 (2015). All 100 mm × 100 mm specimens were preconditioned at 23 ± 2 °C and 50 ± 5% relative humidity. To minimize heat loss, the bottom and sides of each specimen were wrapped in aluminum foil, and a heat flux of 50 kW/m² was applied. Each test was repeated thrice to ensure reproducibility.

Based on the cone calorimeter data, key fire performance indicators were analyzed, including the time to ignition (TTI), total heat release (THR), peak heat release rate (PHRR), and mass loss. These metrics provide a comprehensive evaluation of the thermal response and combustion behavior of the treated plywood under heat exposure.

Mechanical and Physical Properties

The bending strengths of the plywood specimens were evaluated using a 5-ton universal testing machine (Hounsfield H50KS, USA). The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined through three-point bending tests under center-point loading. The specimens were tested in both the longitudinal (0°) and transverse (90°) directions relative to the grain orientation. The crosshead speed was set to 10 mm/min, and the span length was adjusted to 24 times the specimen thickness.

The water-resistant tensile shear strength was measured using the same testing machine. Specimens underwent a cyclic treatment consisting of boiling in water for 4 h, drying at 60 ± 3 °C for 20 h, and boiling again for 4 h. Subsequently, the samples were immersed in room-temperature water to maintain saturated conditions during the testing. Tensile shear tests were performed under bidirectional axial tension at a loading speed of 2 mm/min, and the maximum load was recorded.

RESULTS AND DISCUSSION

Retention Level, Density and Moisture Content Changes

This study investigated changes in the retention level, density, and moisture content of flame retardant-treated larch and pine plywood, which are widely used softwood species in structural applications. These physical property changes are closely related to the subsequent analysis of the combustion performance and mechanical behavior. As shown in Table 3, both species exhibited relatively high retention levels immediately after flame retardant impregnation. However, the observed trends reflect the known differences in treatability and fixation efficiency between the two species, with larch having a lower permeability to aqueous flame retardants. Larch is classified as a refractory or difficult to treat softwood species due to its low permeability, high resin and gum content, and drying difficulties, all of which may limit effective impregnation and fixation of flame retardants (Bao *et al.* 1984).

The change in density was more pronounced in the pine plywood. Its density increased from 0.55 g/cm³ before treatment to 0.82 g/cm³ after impregnation, which was likely due to the retention of solid components from the flame retardant within the wood structure. In contrast, larch plywood exhibited minimal change, maintaining a density of approximately 0.64 g/cm³ regardless of treatment, indicating limited penetration and retention.

The moisture content measurements showed contrasting trends. In larch, the moisture content slightly decreased from 7.85 ± 1.33% (untreated) to 6.69 ± 1.36% after treatment. Conversely, pine plywood exhibited a significant increase from 8.04 ± 0.98% to 13.67 ± 2.61%. This is likely due to the hygroscopic nature of the water-soluble flame retardant retained in the wood matrix.

The species-specific differences in flame retardant retention and moisture-related behavior were closely associated with the trends in fire resistance and mechanical performance, as demonstrated in the following results.

Table 3. Retention Levels of Flame Retardant in Larch and Pine Plywood under Drying Conditions

Species	Post-impregnation (kg/m³)	Air-dried (kg/m³)
Larch	135.8±20.4	112.81±17.9
Pine	692.6±22.4	627.27±46.4

Combustion performance evaluation

The combustion performance of treated plywood, as evaluated using ISO 5660-1 (cone calorimetry) and KS F 2819 (45° flammability test), is summarized in Table 4 and Figs. 2 and 4.

In the cone calorimetry test, the treated larch plywood exhibited moderate improvement, as evidenced by delayed ignition and a slight reduction in heat release. In contrast, pine plywood showed substantial enhancement in all key metrics, particularly in ignition delay and THR. These results are consistent with the higher flame retardant retention observed in pine, supporting a strong correlation between chemical uptake and fire performance. The average flame retardant retention was 627 kg/m³ in pine and 113 kg/m³ in larch, explaining the more effective flame suppression observed in pine. The HRR curves shown in Fig. 2 support these trends. In pine plywood, flame retardant treatment delayed the time to ignition and significantly reduced the heat release rate (HRR). While the untreated pine showed a sharp peak within 50 s, the treated specimen displayed a slower increase and a noticeably lower peak value. This is attributed to the formation of a dense and continuous char layer, which is supported by the generation of B_2O_3 from boron compounds during combustion, which acts as a physical barrier to block oxygen and heat transfer (LeVan and Tran, 1990; Abdalla *et al.* 2003). These findings are also consistent with previous studies reporting that B–O bonds in boron–phenolic systems promote char formation and enhance flame retardancy (Zhang *et al.* 2023; Wang *et al.* 2014). Additional visual evidence is shown in Fig. 3. The surface of the treated pine sample in image (d) appears more compact, uniform, and matte with fewer visible cracks than the untreated sample in image (c). This observation suggests that the treated char layer provides better thermal insulation. In contrast, the larch plywood showed only minor differences before and after treatment.

The results of the 45° flammability test also demonstrate a clear contrast between the two species. In the pine, the untreated specimens displayed a significant flame spread across the surface and a large charred area, as shown in Fig. 4c. Treated specimens, shown in Fig. 4d, exhibited a localized burn pattern with a significantly smaller char area. The difference was less pronounced in the larch samples (Fig. 4a and 4b), consistent with their lower absorption of the flame retardant.

Table 4. Comparison of Combustion and Flammability Performance of Larch and Pine Plywood Before and After Fire Retardant Treatment

Species	Treatment	Fire Performance				Flammability Performance			
		TTI (s)	PHRR (kW/m^2)	THR (MJ/m^2)	Mass loss (%)	After glow time (s)	After flame time (s)	Char length (cm)	Char Area (cm^2)
Larch	Untreated	20-25	139.4	23.8	17.5	8.58	4.93	10.33	51.94
	Treated	35-40	120.6	16.4	14.0	3.25	4.68	10.18	55.12
Pine	Untreated	25	219.3	29.7	42.3	51.50	8.53	17.03	130.16
	Treated	130-150	101.8	7.9	17.9	1.15	1.73	6.05	20.31

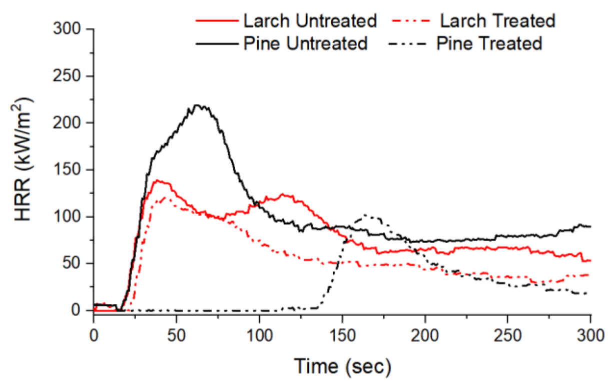

Fig. 2. Heat release rate (HRR) profiles of untreated and flame retardant-treated larch and pine plywood under the conditions of the cone calorimeter test

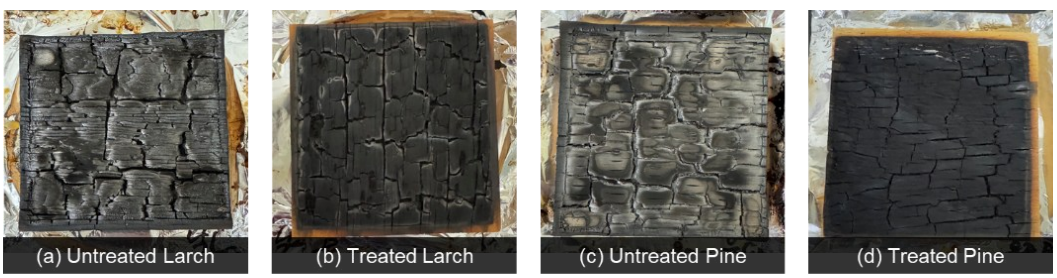

Fig. 3. Surface char formation of larch and pine plywood after the cone calorimeter test: (a) untreated larch, (b) treated larch, (c) untreated pine, (d) treated pine

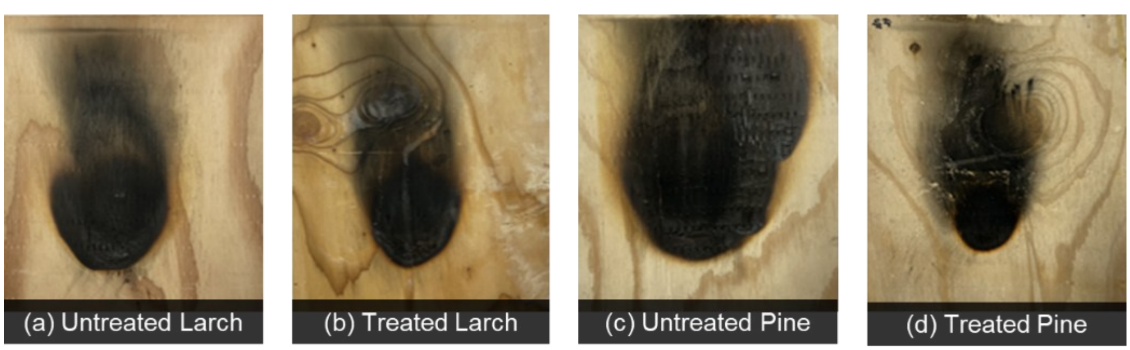

Fig. 4. Surface charred area of larch and pine plywood after the 45° flammability test: (a) untreated larch, (b) treated larch, (c) untreated pine, (d) treated pine

In summary, the treatment enhanced the combustion and flammability of both wood species. However, the improvement in larch has been limited, likely due to its lower permeability and retention, which constrained the effectiveness of the flame retardant mechanism. The pine exhibited consistent and substantial improvements in all key performance metrics, supporting its applicability as a flame retardant wood-based construction material. These findings are in line with Dogan *et al.* (2021), who reported that the incorporation of boron compounds into resin systems contributes to improved flame retardancy and stability in wood-based composites.

Bending Strength

The influence of vacuum pressure impregnation with the composite flame retardant was evaluated in terms of the flexural performance of the structural plywood. Tests were conducted on larch and pine plywood by measuring MOR and MOE in both the longitudinal (0°) and transverse (90°) directions relative to the grain. Independent-sample t-tests were used to analyze the differences between the untreated and treated groups, and the results are presented in Table 5.

In larch plywood, both the MOR and MOE decreased slightly in both directions following treatment. However, these changes were not statistically significant, with p-values exceeding 0.05. This limited effect is likely due to the low average retention level of 19.5 kilograms per cubic meter, which may have restricted the penetration depth and minimized the structural changes in the wood matrix.

In contrast, pine plywood exhibited statistically significant reductions in the MOR and MOE in the longitudinal direction after treatment, with p-values less than 0.01. These decreases are believed to result from the high average retention of 404 kilograms per cubic meter, indicating deeper chemical infiltration that could affect structural cohesion and bond quality. Despite this, the treated pine specimens still met the structural requirements defined by KS F 3113:2021, which stipulate MOR values of at least 30 megapascals and MOE values of at least 5.0 gigapascals in the 0° direction.

In the 90° direction, no statistically significant changes were observed in either the MOR or MOE for either species. However, some pine specimens recorded MOE values below the 4.0 gigapascal threshold. This reduction may be attributed to the localized flame retardant deposition around the knot regions, potentially causing stress concentration or disruption of the adhesive interface. Such effects may reflect the anatomical variability within the wood.

In summary, the mechanical responses to flame retardant treatment varied according to the wood species and chemical retention level. However, the treated plywood maintained its structural integrity when processed under suitable conditions. These findings underscore the need to optimize the impregnation parameters according to the specific anatomical and permeability characteristics of each wood species.

Table 5. MOR and MOE of Plywood at 0° and 90°

Species	Treatment	MOR_0 (MPa)	MOR_{90} (MPa)	MOE_0 (GPa)	MOE_{90} (GPa)
Larch	Untreated	49.1±16.4	31.3±8.7	8.37±0.6	4.96±0.5
	Treated	25.1±4.4	27.4±9.6	6.97±0.8	4.28±0.3
Pine	Untreated	60.2±7.2	27.6±2.9	9.42±0.3	3.04±0.3
	Treated	31.9±3.8	28.1±3.7	7.48±0.6	3.04±0.4

Note: 0° = loading parallel to grain; 90° = loading perpendicular to grain.

Tensile-Shear Strength

As shown in Fig. 5, there were no statistically significant differences in the water-resistant tensile-shear strength between the untreated and treated groups for either larch or pine plywood, with all p-values exceeding 0.05. The average shear strength under all conditions exceeded the minimum requirement of 0.7 MPa specified in KS F 3113. This criterion was met in both normal- and reverse-direction tests. Larch plywood showed nearly identical values between the two groups, suggesting negligible impact of the flame retardant treatment on bonding performance. For pine plywood, a slight increase in the average strength was observed in the treated specimens, although the difference was not statistically significant.

These results suggest that impregnation with the composite flame retardant did not impair adhesive bonding and that the treated plywood continued to meet the structural application standards. These findings support the viability of this treatment method for producing fire-resistant plywood without compromising its mechanical integrity.

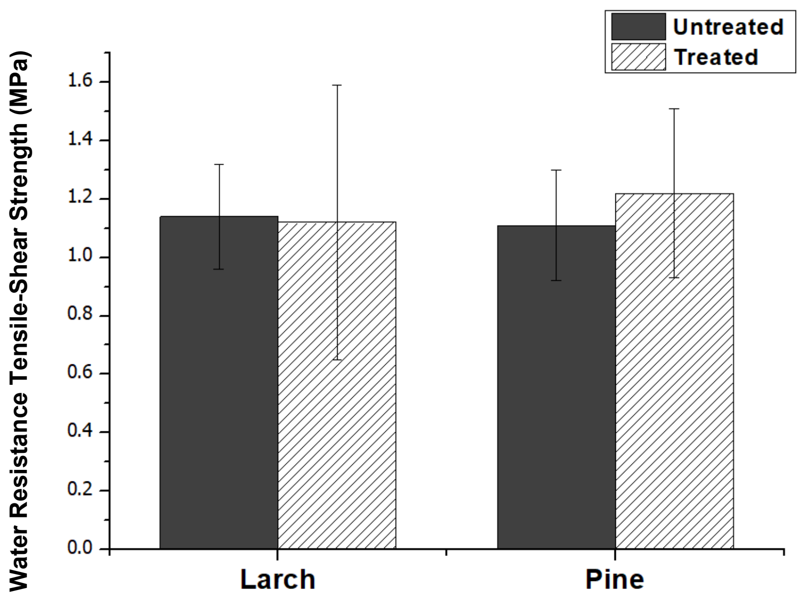

Fig. 5. Water resistance tensile-shear strength of untreated and flame retardant-treated larch and pine plywood

In summary, the PBAX flame retardant demonstrated excellent impregnation, fire resistance, mechanical strength, and adhesive stability in structural plywood, confirming its practical viability. Optimization of treatment parameters according to species-specific characteristics and permeability may further enhance its commercial applicability. Future research should expand the application of PBAX to a wider range of structural wood products and establish appropriate treatment conditions for each product type to systematically evaluate their properties and performance.

CONCLUSIONS

This study developed a boron-based composite flame retardant (PBAX). This was done by combining a low-molar-ratio phenol–formaldehyde resin with a highly concentrated boric acid–borax solution and applied it to structural plywood using a full-cell vacuum pressure process. Through evaluating the impregnation behavior, fire resistance, and mechanical properties of the treated larch and pine specimens, the study demonstrated the following key outcomes.

1. Impregnation experiments demonstrated that PBAX could be reliably applied to structural plywood *via* vacuum pressure treatment. Although the retention varied by species, effective penetration was achieved in both cases. Pine exhibited a high average retention of 627 kg/m³ and a notable density increase from 0.55 to 0.82 g/cm³, whereas larch showed a lower retention level of 113 kg/m³ with negligible changes in density.
2. The evaluation of the mechanical performance revealed that larch plywood exhibited minimal differences in bending strength (MOR) and modulus of elasticity (MOE) before and after treatment. In pine, a significant reduction was observed in the 0° direction, yet all specimens met the KS F 3113 standard requirements (MOR ≥ 30 MPa, MOE ≥ 5.0 GPa). However, the MOE in the 90° direction for some pine specimens fell below the 4.0 GPa threshold, likely due to stress concentrations near knot areas.
3. Tensile-shear strength analysis showed no statistically significant differences between the treated and untreated groups for either species. All specimens exceeded the minimum KS requirement (0.7 MPa), indicating that PBAX impregnation did not adversely affect the adhesive bonding performance.

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