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## Evaluating the mechanical and fire-resistance properties of modified fast-growing Chinese fir timber with boric-phenol-formaldehyde resin



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### HIGHLIGHTS

- The activation energy of boric-containing PF was higher than that of common PF.
- High-frequency heating provided a rapid rise in temperature for BPF-specimen.
- The strength grade of modified wood with BPF resin was increased greatly.
- The fire performance of modified wood with BPF resin was improved significantly.

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### ABSTRACT

The abundant fast-growing Chinese fir was confined to nonstructural use for its poor mechanical and fire performance, and classified as low-quality building materials. Herein, Chinese fir was impregnated with boric-phenol-formaldehyde (BPF) resin, and then processed using high-frequency heating according to practical processing and application. The properties of BPF-specimen were tested, and the results showed, compared with the common plate hot pressing, high-frequency heating was an efficient technology for its high heating rate according to commercial process. Compared with the control, the mechanical properties and fire performance of BPF-specimen were improved obviously, and the more the resin retain, the better the properties of the modified specimen.

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### 1. Introduction

In recent years, timber structure has been gaining more attention due to its friendly environment and appearance attractions. Wood materials are not fire-resistant. The decrease of mechanical properties of wood exposed to fire often limits its practical applications.

Wood thermal degradation produced a charred layer on surfaces and diminishes the effective cross-sectional area [1]. The char layer is a good thermal insulator and protects the interior cross-section [2]. From the temperature distribution, the gradient of fire-exposed wood is large due to the low thermal conductivity of the charred layer [3]. Therefore, fire resistance of large cross-

section timber member like glued laminated timber (glulam) and cross laminated timber (CLT) exhibit good high-temperature stability.

The experimental results showed the mechanical properties of Rowan wood at the high temperature were lower than the normal temperature [4]. The color dimming of wood specimens occurred within the first 4 h, and bending strength decreased by 44–50% under high temperature conditions [5,6]. In addition to the above mentioned, the following should be gained more concerns that wood burn on its surface, release combustible energy (heat and combustible gas), would be decomposed and thus contribute to fire propagation accompanied by a loss in mass when exposed to fire or high temperature. The applications of wood materials were strictly limited in construction, especially as used as structural members. Therefore, enough fire safety is essential for structural wood materials in cases where a higher level of fire performance is desire in timber structure.

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The most common fire retardant agents used for wood materials are inorganic salts, boron compounds, phosphoric acid, monoammonium phosphate, diammonium phosphate, ammonium sulfate, nitrogen, and zinc chloride [7–10]. In case of fire accident, ideal fire retardants can delay the ignition and the flame spread. They can also reduce the rate of heat release [11]. Wood-based panels processed with urea formaldehyde resin added boric acid and borax showed that burning resistance increased. However, their bending strengths decreased [12]. Wood treated with the inorganic salts is more hygroscopic [7].

The boron compounds are the safest in current use with no harmful effects [13]. Boric acid had been applied as most components in many fire retardants. It was also found that boron compounds could reduce the flame spread of wood-based materials, and was highly efficient for preventing glowing combustion [10,14–15]. Conclusion can be obtained that boric acid-based fire retardants could be used to be improve the fire resistance of wood materials, but the reduction in mechanical properties of treated wood was found with addition of fire retardants [16,17].

Thermosetting phenolic resins have excellent mechanical and thermal properties, and are widely used as thermal insulation, fire retardant and ablative materials [18–21]. So, phenolic resin may be used as a substitute for traditional wood fire modifier. However, its oxidation resistance under high temperature conditions needed improving furtherly for the application in refractory materials [22]. Boron was found to exhibit excellent oxidation resistance [23–26]. Boron-containing phenolic resin used as a novel modifier would be possible to improve fire performance of wood elements in timber structure.

Fast-growing Chinese fir is widely planted in China. It is widely used as furniture and structural material. Chinese fir can be modified with phenol-formaldehyde resin modified with boric acid (BPF) resin to improve its mechanical properties and fire performance.

The primary objectives of this study were to evaluate the mechanical and fire-resistance properties of modified fast-growing Chinese fir timber with boric-phenol-formaldehyde resin.

## 2. Materials and methods

### 2.1. Materials

Fast-growing Chinese fir was chosen randomly from timber suppliers in Jiangsu province, south eastern China. It was dried to moisture content from 8% to 12%. The air-dry density of CF was  $0.355 \text{ g}\cdot\text{cm}^{-3}$  at 12% moisture content. The knotless, non-crack and normally grown Chinese fir timber was selected as test specimens.

Formaldehyde (38.5%) was purchased from Xilong Chemicals Co., Ltd. (Guangdong, China). The additives, such as, sodium hydroxide (96%), boric acid (99.5%) and phenol (99%) were supplied by the Shanghai Shiyi Chemical Reagent Co., Ltd., Shanghai, China. Boric-phenol-formaldehyde (BPF) resin was synthesized according the description by Wang [27]. 148.21 g of 38.5% formaldehyde solution and 95.06 g phenol were slowly added into a round-bottomed flask, respectively, and then heated to 40 °C in water-bath. After 30.00 g of 40% sodium hydroxide was added into the reaction solution, the reaction solution was mixed for 15 min using a mechanical stirring. The reaction solution was heated to 75 °C under continuous mechanical stirring for 45 min. Then 18.64 g boric acid was added and the solution was stirred for another 15 min at 95 °C. Finally, the mixture was cooled to below 40 °C and the resin was then stored.

Commercial phenol-formaldehyde (PF) resin was acquired from plywood supplier in Jiangsu province, P.R. China. The original solid contents of BPF and PF resins were 49.3% and 52.79%, and their pH values were 7.9 and 10.1, respectively. Both resins were stored at a room with temperature between 0 °C and 5 °C to slow the curing rate. Before each use, the resins were homogenized for 5 min using a mixer running at a pumping capacity of  $200 \text{ L}\cdot\text{min}^{-1}$ .

### 2.2. Wood modification

BPF resin was diluted and homogenized to 20%, 30% and 40% concentrations. The specimens with dimensions of 38 by 89 by  $1000 \text{ mm}^3$  were impregnated with different concentrations of BPF resin in a high-pressure steel sealed capsule. The specimens were subjected to a 15 kPa vacuum for 4 h, and then soaked in BPF resin

at 450 kPa and 50 °C for 5 h. The following step was the removal of excess resin under a 15 kPa vacuum for another half an hour. After treatment, the specimens were turned into the next step of moisture content adjustment. The specimens with the resin were dried in an oven at 60 °C for 10–12 h to moisture content of 15–20%. The treated specimens were heated with the press (XLB universal machine made in Qingdao city, China), and high-frequency (CGYJ-150D high frequency generator made in Shijiazhuang city, China) to allow the polymerization. The specimens were cut into test pieces. test pieces were conditioned at  $(20 \pm 2) \text{ }^\circ\text{C}$  and  $(65 \pm 5)\%$  relative humidity until the mass was stabilized.

### 2.3. Temperature distribution measurement during resin polymerization

In the process of resin polymerization, K-type thermocouples (Shanghai Xinghui Automation Instrument Factory, China) were used to measure temperature distribution in wood specimens using common plate hot processing. The temperatures were measured using a DX1012 temperature patrol measure meter (Yokogawa, Japan). Temperature was recorded every minute.

In high-frequency heating, temperature distribution in BPF-containing wood specimens was measured with a FOTS-CTM-300A optical fiber temperature sensor system (made in Beijing city, China). The frequency and the plate voltage was set 6.78 MHz and 5 kV, respectively. Temperature was recorded every five seconds.

The schematic of thermocouples located in specimens was shown in Fig. 1.

In Fig. 1, the average temperature at the four points, 1, 1', 3 and 3' was set as the surface temperature which was 5 mm away from the wood specimen surface. The average temperatures at points 2, 2', 4 and 4' was set as the central temperature at 1/2 h.

### 2.4. Tests for mechanical properties

The specimens for static bending strength testing were  $20 \text{ mm} \times 20 \text{ mm} \times 300 \text{ mm}$ , and those for compressive strength were  $20 \text{ mm} \times 20 \text{ mm} \times 30 \text{ mm}$  (Tangential, radial, and longitudinal directions). The bending strength ( $\sigma_b$ ) and compressive strength ( $\sigma_c$ ) were determined using the following formulas [28,29]:

$$\sigma_b = 3 \cdot P_{max} \cdot l / (2 \cdot b \cdot h^2) \quad (1)$$

$$\sigma_c = P_{max} / (b \cdot h) \quad (2)$$

where,  $\sigma_b$  was bending strength (MPa);  $\sigma_c$  was compressive strength (MPa);  $P_{max}$  was ultimate bearing capacity (N);  $l$ ,  $b$  and  $h$  were the span, width and thickness of tested specimens (mm), respectively.

The bending and compressive strengths were conducted using a universal wood-testing machine with a 50-N load cell. The speeds of load were set 3 and  $1 \text{ mm}\cdot\text{min}^{-1}$  in bending and compressive tests, respectively. Fifteen replicates were performed for each condition.

### 2.5. Determination for curing kinetics of boric-phenol-formaldehyde resin

The parameters of curing kinetics for BRF was characterized using differential scanning calorimetry (DSC) method (NETZSH DSC200F3, Germany). Vacuum dried resin at 20 °C temperature was grinded, and tested under nitrogen atmosphere. The heating rates were 5, 10, 15 and  $20 \text{ K}\cdot\text{min}^{-1}$ , respectively, and scanning temperature was set from 20 to 220 °C at the  $30.0 \text{ mL}\cdot\text{min}^{-1}$  nitrogen flow rate.

Kissinger method [31] was applied to analyze the curing characteristics of BPF according to the formula:

$$\ln(\beta/T_p^2) = \ln(A \cdot R/\Delta E) - \Delta E/(R \cdot T_p) \quad (3)$$

where,  $T_p$  was the absolute peak temperature (K),  $\beta$  was the heating rate ( $\text{K}\cdot\text{min}^{-1}$ ),  $\Delta E$  was the activation energy ( $\text{kJ}\cdot\text{mol}^{-1}$ ),  $A$  was pre-exponential factor ( $\text{s}^{-1}$ ), and  $R$  was the universal gas constant ( $8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ ).

According to the Kissinger differential equation. The slope of the curve can be obtained after linear fitted. The total activation energy  $\Delta E$  of BPF was obtained from the curves of heat flow rate and temperature by the method of Kissinger.

### 2.6. Tests and analysis for fire performance

The fire performance, including heat release rate, total heat release, total smoke release, mass retain and residual cross section of wood specimens were carried out according to ISO5660-1 [32] using FTT0007 cone calorimeter (Fire and Combustion Characteristics Testing Technology Co., Ltd, China) (Fig. 2).

All the thermal tests were conducted by placing the specimens in a sample holder in a horizontal position. The back of the samples was coated with low-conductivity material to reduce heat losses to the sample holder. The heat flux was set  $50 \text{ kW}\cdot\text{m}^{-2}$ , and the interval of data acquisition was one second.

Two specimens were tested in each group, and the average values were used.

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