



## Fire retardancy of an aqueous, intumescent, and translucent wood varnish based on guanlyurea phosphate and melamine-urea-formaldehyde resin



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### ABSTRACT

Various concentrations of guanlyurea phosphate (GUP) were incorporated into an aqueous melamine-urea-formaldehyde (MUF) resin to synthesize a fire-retardant, translucent, and intumescent varnish. The effects of the GUP on the curing, light transmittance, and thermal stability of the resulting varnish, and combustion behavior of the varnish-coated plywood were determined by viscosimetry, Fourier-transform infrared (FT-IR) spectroscopy, ultraviolet-visible (UV-vis) spectroscopy, thermogravimetry (TG), and cone calorimetry, respectively. The results show that GUP decelerated the curing rate; the resulting varnish film was translucent and UV-absorbing. The calorimetry demonstrated that GUP did not change the thermal stability but significantly facilitated charcoal formation of the varnish. The presence of the GUP resulted in an increased intumescent thickness of the varnish and substantially suppressed and delayed the mass loss, smoke production, and heat release of the coated plywood. At 12% GUP concentration the resulting varnish film exhibited an optimal combination of translucency and fire retardancy. These findings demonstrate that the GUP can be incorporated into aqueous MUF resin for the synthesis of a clear intumescent varnish capable of fire retardancy.

### 1. Introduction

Wood is a building material and decorative material, and exhibits unique esthetic and sustainable characteristics; however, wood is flammable, which represents a considerable risk. Therefore, it is common to treat wood with fire retardants via full impregnation [1,2]. The advantage of the impregnation treatment is that the fire retardant can diffuse into the wood, thereby providing full and uniform protection. Common fire-retardant agents include phosphate, carbamide, and borate, either individually or synergistically. Guanlyurea phosphate (GUP), which is synthesized with dicyandiamide and phosphoric acid (PA), was shown to be highly effective as a fire retardant in wood to resist combustion and smoke release [3]. The solubility of GUP is limited in water (< 12%) and it is commonly combined with boric acid (BA) as a wood fire retardant [4]. The GUP/BA mixture is dissolved in water and vacuum/pressure impregnation is used to deposit them in wood, impart a fire resistance. The wood thus treated exhibits a considerable reduction in smoke production and heat release and increases in charcoal residues. The challenge is that it requires a relatively high load (thereby high cost) and that leachability from the wood is high [1]. The unreacted PA in the mixture can catalyze the hydrolysis of

polysaccharides, resulting in the reduction in mechanical strength of the wood [4]. In addition, the impregnated wood requires a cost- and energy-intensive drying process.

An alternate strategy is the application of intumescent fire-retardant coatings to isolate the wood from the fire [5,6]. Coating is a relatively simple process and the application efficacy is high compared to impregnation treatments with fire retardants. The intumescent fire-retardant coatings generally contain polymeric binders, fire-retardants, polyhydroxy (carbon donors), blowing agents, and additives [7,8]. Most of fire retardant compositions used in the coatings are the phosphates [9,10]; other compositions, such as montmorillonite, titanium oxide, and silica, were also reported to be used for improving the fire retardancy [11–13]. Intumescent coatings have been developed and used extensively for wood-and steel-framed constructions [14,15]. Such coatings protect wood from fire principally based on the following mechanisms: 1) During the pyrolysis process, the coatings melt or decompose to molten substance and the blowing agent releases incombustible gases. The gases are entrapped in the molten layer, resulting in volumetric intumescence; 2) The fire retardants in coatings are pyrolyzed to produce inorganic acids that catalyze the carbon donor and form porous and compact charcoal [14,16]. The porous charcoal

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functions as a barrier to heat and O<sub>2</sub>. Current challenges in the use of intumescent fire-retardant coatings include finding alternatives to the use of organic solvents, making the fire-retardant compounds compatible with the polymeric binder, and making the final coatings translucent on the wood surface [17].

The objective of this study is to formulate an intumescent fire-retardant varnish by incorporating the limited water-soluble GUP in melamine-urea-formaldehyde (MUF) resin. The effect of the GUP concentration on the curing reaction and transmittance of the resulting varnish films were determined by Fourier-transform infrared (FT-IR) spectroscopy and ultraviolet-visible (UV-vis) spectrophotometry, respectively. The thermal stability of the varnish film and the combustive behavior of the varnish-coated plywood were investigated by thermogravimetric (TG) analysis and cone calorimetry, respectively.

## 2. Materials and methods

### 2.1. Materials and chemicals

Plywood made from poplar (*Populus adenopoda* Maxim.) wood veneers was cut into panels, measuring 100 mm × 100 mm × 3.5 mm (length × width × thickness) and served as the substrate for the varnishes. Pentaerythritol (PER) and PA with 85% solid content were purchased from Yaohua Chemical Co. Ltd. (Tianjin, China). The MUF resin in the form of a powder was provided by the Debon Adhesive Co. Ltd. (Harbin, China). The GUP was synthesized in the Key Laboratory of Bio-based Material Science and Technology (Ministry of Education, China).

### 2.2. Varnish preparation and application on the plywood

The PER (carbon donor), GUP (fire retardant), MUF (binder and blowing agent) and water (solvent) were mixed and homogenized in a ball-grinding mill at 90 r min<sup>-1</sup> for 90 min. Subsequently, the PA (catalyst) was added to the homogenized solution under stirring until the pH value dropped to 1.5 (target varnish). The concentrations of PER (10%) and MUF (15%) remained constant in the system; the concentrations of GUP were 0, 4, 8, 12, 16, and 20%. The resulting varnishes were termed V<sub>GUP0</sub>, V<sub>GUP4</sub>, V<sub>GUP8</sub>, V<sub>GUP12</sub>, V<sub>GUP16</sub>, and V<sub>GUP20</sub>, respectively (Table 1). However, the varnishes containing 16 and 20% GUP (V<sub>GUP16</sub> and V<sub>GUP20</sub>) were opaque due to the limited solubility of GUP. Therefore, these two varnish formulas were given up in this study. A single surface of the plywood board was brushed with a layer of the prepared varnish (0.1 g cm<sup>-2</sup>). The coated board was then stored under ambient conditions for one week to allow the varnish to dry completely. The thickness of the dry varnish film was measured using a light microscope (Nikon eclipse e600 equipped with a Nikon digital camera DXM 1200, Nikon Instruments Inc., NY, USA) and was 300 μm on average.

**Table 1**

The composition formulations of varnishes prepared in this study (wt%).

Abbreviation	GUP	MUF	PER	PA <sup>a</sup>	Remarks
V <sub>GUP0</sub>	0	15	10	ND	Opaque emulsion
V <sub>GUP4</sub>	4	15	10	ND	Translucent emulsion
V <sub>GUP8</sub>	8	15	10	ND	Translucent solution
V <sub>GUP12</sub>	12	15	10	ND	Translucent solution
V <sub>GUP16</sub>	16	15	10	ND	Opaque emulsion with insoluble particles
V <sub>GUP20</sub>	20	15	10	ND	Opaque emulsion with insoluble particles

<sup>a</sup> The phosphoric acid (PA) is used to adjust the pH value of target varnish to 1.5 and the amount of PA is not numerical.

### 2.3. Viscosity determination

After preparation, the fresh varnish was immediately tested using a rotational viscometer (NDJ-8S, Fangrui Instruments Co. Ltd., Shanghai), with a rotation speed of 30 r min<sup>-1</sup>. The dynamic viscosity of the specific varnish was measured together with the aging time. The test was stopped when the viscosity of the varnish reached 140 mPa s (end viscosity) to avoid any damage of the viscometer by excessively high viscosity. The time it took from start viscosity to end viscosity was recorded.

### 2.4. FT-IR spectroscopy

The V<sub>GUP12</sub> was applied on a glass plate (0.1 g cm<sup>-2</sup>) and dried under ambient conditions. The infrared spectra of the V<sub>GUP12</sub> were recorded using an FT-IR spectrometer equipped with an attenuated total reflection (ATR) attachment (Frontier, PerkinElmer Inc. Akron, OH, USA) after specific drying periods. In addition, the wood charcoals carefully separated from the combusted varnish-coated plywoods after cone calorimetric test were also measured using FTIR. The spectra were conducted using 40 scans and at 4 cm<sup>-1</sup> resolutions at room temperature at wavenumbers ranging from 400 to 4000 cm<sup>-1</sup>.

### 2.5. UV-vis spectrophotometry

The prepared varnish was poured onto a highly polished silicon chip and evenly spread on the chip surface using a glass rod (0.1 g cm<sup>-2</sup>). The coated silicon chip was dried under ambient conditions and the light transmittance was measured at the wavelength ranging from 200 to 800 nm using a UV-vis spectrophotometer (TU-1901, Shanghai Spectrometers Co. Ltd., Shanghai, China).

### 2.6. TG analysis

The GUP, MUF, and varnish films (each 10 mg) were placed in platinum crucibles to determine their thermal decomposition using the TG instrument TGA4000 (PerkinElmer Inc. Akron, OH, USA). The test was performed in a nitrogen atmosphere with a purge gas flow of 50 ml min<sup>-1</sup>. The temperature was increased from 30 to 800 °C at a heating rate of 10 °C min<sup>-1</sup>. The measurements were conducted in triplicate for each sample. The TG curves of the sample and their derivative curves (DTG) were recorded through the tests.

### 2.7. Cone calorimetric analysis

The combustion properties of the plywood coated with the prepared varnishes were tested using a cone calorimeter (Fire Testing Technology Ltd, East Grinstead, UK) according to the standard ISO 5660-1 (2002). The sides and rear surfaces of the specimens were covered with aluminum foil, leaving the coated surfaces exposed to the fire. The test was run at a heat flux of 50 kW m<sup>-2</sup>. The combustion parameters, such as residual mass (charcoal), heat release, smoke production, and carbon oxide concentration, were recorded automatically during the test. The thickness of the intumescent varnish film on the burnt samples was measured using a vernier caliper. The test was conducted in triplicate for each sample treatment.

### 2.8. X-ray photoelectron spectrometric analysis

The charcoal of varnishes and plywood, separated from the coated samples after the cone calorimetric test, were measured at room temperature using an X-ray photoelectron spectrometer (XPS) equipped with monochromatic AlKα radiation (1486.6 eV) (K-Alpha™, Thermo Fisher Scientific Co., Ltd, Shanghai, China). The X-ray beam was a 100 W- and 200 mm diameter-beam raster over a 2 mm by 0.4 mm area on the sample. High-energy photoemission spectra were collected using

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