



# Combined effects of ammonium polyphosphate and talc on the fire and mechanical properties of epoxy/glass fabric composites



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## ABSTRACT

The combined effects of ammonium polyphosphate and talc on the fire and mechanical behaviour of epoxy/glass fabric composite systems and neat epoxy resin were investigated. Recorded data from cone calorimeter and UL-94 vertical burning experiments reveal that the incorporation of additives can enhance the flame retardancy of both epoxy resin and glass fabric reinforced composites. TGA results demonstrate that the introduction of additives improve the thermal stability of epoxy resin at high temperatures. Moreover, the addition of flame retardants to epoxy resin increases the tensile and flexural moduli. However, due to the incompatibility of the additives with epoxy, the strengths are compromised. Then again, the epoxy/glass fabric composites with flame retardant additives have improved the fire properties without significantly affecting the mechanical performance.

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## 1. Introduction

Epoxy resin has been used in wide range of applications, such as composites, coatings, adhesives and electrical devices, due to its properties, namely, excellent chemical and solvent resistance, high toughness and adhesiveness, high strength to weight ratio and dimensional stability [1–3]. However, flammable characteristics of epoxy can restrict its extensive application where high flame resistance is required [4,5], and it is important to develop the flame retardance in neat epoxy products and composites based on it.

An incorporation of flame retardant (FR) additives, which are generally categorized as halogen free and halogenated FRs, is an effective and common method to enhance the flame retardancy of epoxy resin [6–8]. The production of toxic gases and corrosive substances, during combustion, is the major drawback of halogenated FRs. Therefore, in recent years researchers have focused on the application of halogen free FR additives due to the environmental concerns [9,10]. There are several types of halogen free FRs, namely, metal hydrates, nitrogen containing FRs, phosphorous based FRs and intumescent systems [11,12]. Metal hydroxides, for instance, magnesium and aluminium hydroxides, provide effective flame retardancy for polymer systems due to the endothermic

dehydration when exposed to heat and fire. Hao et al. [10] have shown that the addition of 35–55 wt% of magnesium hydroxide can significantly reduce the flammability of unsaturated polyester resin. However, a high loading level of metal hydroxides to reach suitable flame retardancy can unfavourably decrease the mechanical properties of the polymer matrices [13–16]. In recent years, there has been an increasing interest in the application of intumescent FRs due to their advantages, including their ability to form foamed char and produce less smoke and toxic gases during combustion [17–20]. Ammonium polyphosphate (APP) is a nitrogen and phosphorous containing halogen free FR which can act as an intumescent system [21]. A considerable amount of literature has been focused on the flame retardant, thermal and mechanical properties of epoxy resin and composites modified with APP [1–5,7]. During the combustion process, APP yields a protective foamed char on the surface of the polymer which can limit the heat and mass transfer between the flame and the underlying materials. As a result, the formation of the intumescent char can effectively reduce the flammability of the polymer [22–25].

Talc is a natural inert filler and is characterised as hydrated magnesium silicate sheet, with the chemical formula  $Mg_3Si_4O_{10}(OH)_2$ . It is not flammable and has the potential to impart flame resistance to polymers. The combination of APP and talc as FR additives has the capability to provide suitable flame retardant properties for thermoplastic polymers. Lechvik et al. [26] have found that the flame retardancy of polyamide 6, containing APP,

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improves by the addition of talc due to the role of talc to enhance the protective properties of the intumescent formed char by the interaction between the polymer and APP. It has also been shown that the incorporation of talc and calcium carbonate can lead to an improvement of fire properties of polypropylene/APP/polyamide 6 system [26]. In a previous patent by our group [27], fire retardant polypropylene including APP and talc has been investigated, exhibiting suitable flame retardant and mechanical properties. In contrast, Duquesne et al. [24] have observed a reduction in the flame retardancy of polypropylene/APP system by adding talc due to inadequate ratio of APP and talc in the system.

To the best of our knowledge, notwithstanding the research done with thermoplastic polymers, little has been reported on the effects of APP and talc on thermoset polymer (e.g. epoxy) based composites in regards to their fire and mechanical properties. Hence the primary objective of this study is to investigate the effects of APP and talc as FRs, according to the proprietary formulations [27], on the flame retardant and mechanical properties of epoxy resin and epoxy/glass fabric composites. The flame retarded epoxy samples were prepared and subjected to cone calorimeter test, UL-94 vertical burning test and mechanical tests (tensile, Flexural, compressive and short beam strength), the results have been compared to those of reference samples without any additives.

## 2. Experimental details

### 2.1. Materials

Diglycidyl ether of bisphenol A (DGEBA) epoxy resin (105 West System epoxy) and polyamine curing agent (206 slow hardener) were provided by Adhesive Technologies Ltd. (New Zealand). Melamine coated ammonium polyphosphate (APP, FR CROS 40) was supplied by Budenheim (Germany). Talc (PlusTalc N625) and antioxidant (Irganox 1010) were provided by Mondo Minerals and BASF, respectively. E-glass fabric (2 × 2 twill weave, 285 g/m<sup>2</sup> areal density) was purchased from Colan (Australia).

### 2.2. Sample preparation

#### Epoxy composites

First, APP and talc were dried in an oven at 75 °C for 10 h to reduce the moisture content. For preparation of samples, containing APP/talc/antioxidant, the required amount of talc was premixed with epoxy resin prior to the addition of APP and antioxidant. It minimised the trapping of APP particles between the silica plates of talc which might lead to deactivation of APP particles. After that, the total weight of 200 g of epoxy and additives were mixed using a high shear mixer (Silverson L4R, USA) at 8000 rpm for approximately 5 min, until a homogenous mixture was obtained. In the case of samples containing APP and antioxidant, the particles of APP and antioxidant were dispersed in epoxy resin using the same method. The hardener was added to the homogenous mixture of epoxy resin and additives (mix ratio of resin to hardener by weight was 5.36:1) and stirred rapidly; then the mixture was degassed in a vacuum chamber at room temperature (21 °C) for 5 min, to remove the air bubbles. Finally, the epoxy resin containing additives and hardener was poured into the silicone moulds with the appropriate dimensions for different tests and cured at room temperature for 72 h. Neat epoxy resin samples were prepared as control specimens. The neat epoxy resin, epoxy containing APP and APP/talc were labelled EP, EPAPP and EPAPPT, respectively.

#### Reinforced epoxy laminates

Eight layers of glass fabric were impregnated with epoxy resin

and epoxy containing additives using hand lay-up method to prepare the reference and flame retarded epoxy laminates, respectively. Individual fabric layers were stacked and laid up after resin impregnation. The impregnated and laid up fabric layers were pressed for curing at 0.7 MPa pressure and room temperature (21 °C) for 72 h, resulting in glass fabric reinforced epoxy resin composites with an average thickness of 2.5 mm. The glass fabric volume fraction was kept at 35.6% ± 2% in all composite samples. The fibre reinforced epoxy (reference), epoxy laminates filled with APP and APP/talc were labelled EPF, EPFAPP and EPFAPPT, respectively. The compositions of epoxy resin composites are listed in Table 1.

### 2.3. Characterisation

#### Cone calorimeter test

Cone calorimeter tests were performed to measure fire reaction properties of epoxy resin samples according to ASTM E1354. All the samples were conditioned in a chamber at 23 °C and 50% relative humidity for 48 h before exposing to 50 kW/m<sup>2</sup> heat flux in horizontal position of a cone heater using a cone calorimeter (FTT, East Grinstead, UK). Square (100 mm × 100 mm) epoxy resin and composites were tested in 2.5 mm thickness. The average values of three replicated tests are reported subsequently. A digital camera (HC-V720 M, Panasonic, Japan) was used to collect the photographs of the chars after cone calorimeter experiments.

#### Fourier Transform Infrared spectroscopy

Fourier Transform Infrared (FTIR) spectra of chars, after cone calorimeter tests, were recorded between 4000 and 500 cm<sup>-1</sup> using a Nicolet FTIR iS 50 spectrophotometer.

#### UL-94 vertical burning test

The UL-94 vertical burning tests were carried out to evaluate the flammability of epoxy resin composites according to ASTM D3801. The flammability of the vertically placed rectangular samples (125 mm length L × 13 mm width W × 2.5 mm thickness t) was measured by two visible flame applications on the bottom of the samples. The samples were categorized into V-0 (flame extinguishment within 10 s without dripping), V-1 (flame extinguishment within 30 s without dripping) and V-2 (flame extinguishment within 30 s with dripping) ratings and also NR (no rating) according to the required time periods for the flame to extinguish, dripping and glowing.

#### Scanning electron microscopy

The morphology of the residues after cone calorimeter tests and the fractured cross-section of composites after tensile tests were analysed using an environmental scanning electron microscope (ESEM, FEI Quanta 200F, USA). All the samples were coated with platinum prior to the microscopic analysis.

#### Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed to characterise the thermal stability (the resistance of a substance against thermal decomposition) of epoxy resin samples using a TGA Q5000 instrument. 3–5 mg samples were inserted into high temperature platinum pan and heated from room temperature to 800 °C at a constant heating rate of 10 °C/min in nitrogen atmosphere.

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