



# Interfacial charring method to overcome the wicking action in glass fiber-reinforced polypropylene composite



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## ABSTRACT

Glass fiber (GF)-reinforced polypropylene composites show high flammability due to wicking actions, which results in accelerated flow of the polymer melt along the GF surface to the flame zone. The conventional bulk charring mode, in which high loading of flame retardants are evenly distributed in the bulk matrix to catalyze the resin into an integrate char-barrier so that the entire composite surface could be covered, is quite inefficient and uneconomic. In this research, an interfacial charring mode differing from the bulk one has been proposed to solve the challenge. The acid-source flame retardants grafted on the GF surface can rapidly carbonize the interfacial resin to form a char layer, which can locally encapsulate the GF during combustion, thus converting the original smooth and polar GF surface to rough and inert carbonates-covered one and preventing the polymer melts from wetting, spreading and flowing on it. In this manner, it effectively eliminated thermodynamic and dynamic factors that cause wicking actions and remarkably improved flame retardance, even at a much lower flame retardant concentration compared with the bulk mode. In addition, without the stress concentration effect caused by the flame retardant particles directly distributed in the bulk resin, the interfacial flame retardant system also showed much better mechanical properties than the bulk one.

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## 1. Introduction

Polypropylene (PP) is an important commodity plastic with good properties and low cost. In practical engineering applications, GF-reinforced PP composites are more widely used due to greatly improved strength, rigidity, heat distortion temperature compared with neat PP [1,2]. As a polymer only composed of C and H elements, flammability is an obvious shortcoming of PP. Therefore, flame-retarded modification for this material is significant either in industries or academia [3–6]. At present, some halogen-free flame retardants, such as phosphorus-containing flame retardants (ammonium polyphosphate [7,8], melamine pyrophosphate [9–12], magnesium hydrate [13], etc.), have been commercially applied in neat PP in the replace of traditional environment-unfriendly halogen ones. However, preparing an efficient halogen-free flame retardant GF-reinforced PP is still a problem because of the wicking actions caused by GF. As is well known,

wicking actions are typical physicochemical phenomena existing in most GF/thermoplastic composites. During the combustion of a composite, although the GFs (wicks) filled in the resin matrix do not flame, the polymer melt (fuel) adsorbs, wets and flows along the GF surface to the fire zone, which evidently accelerates the conveying speed of the fuel and further intensifies the combustion, therefore making the composites much more difficultly flame-retarded than neat PP [14,15].

Currently, bulk charring in condensed phase is acknowledged as the most efficient halogen-free flame resistance mode for PP or PP composites [16–18]. In this mode, some acid-source flame retardants are directly added and compounded with PP melt, and finally evenly distributed in the bulk resin. After the combustion occurs, the chemical interaction of the resin and the released acids from the flame retardants can form a protective foamed char layer covering the entire surface of the burning materials to result in quick self-extinguishment by isolating the oxygen, volatiles and heat [19]. Certainly, only quickly produced continuous and compact char layer can endow the material with good insulation [20–23]. The construction of the above char morphology generally relies on a great amount of flame retardants loaded. Unfortunately, present

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research show that the bulk charring mode is actually inefficient for PP/GF system even with a very high loading flame retardant. This can be explained as that the wicking actions give rise to a much higher flame rate for PP/GF composite, accordingly the fire rapidly extends to the material interior before the formation of an isolation char layer.

## 2. Experimental

### 2.1. Materials

Polypropylene (PP) with a melt index of 3.0 g/10 min was purchased from Balin Petroleum and Chemical Subsidiary Company, Petroleum and Chemical Co., China. Raw glass fibers (RGF) with a diameter of 10  $\mu\text{m}$ , were provided by Jushi Group Co., China. Raw glass sheets (RGS) with  $1 \times 3 \times 4 \text{ mm}^3$  were purchased from Sitong Chemical instruments Co., China. Reactive flame retardant, 9, 10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) was purchased from Shengshida Tech & Trade Co. Ltd., China. Silane coupling agent, vinyl-tri-(2-methoxyethoxy)-silane (A172), transparent liquid, was purchased from Chengdu Thinkbond Chemical Co. Ltd., China.

### 2.2. Preparation of the glass fibers and sheets grafted by the flame retardants

DOPO powder and A-172 liquid with the same mole ratio were mixed at 120 °C. The reagents that DOPO dissolved in A-172, were poured into a three-necked flask equipped with an oil-bath pot, a mechanical stirrer and a reflux condenser. The reaction

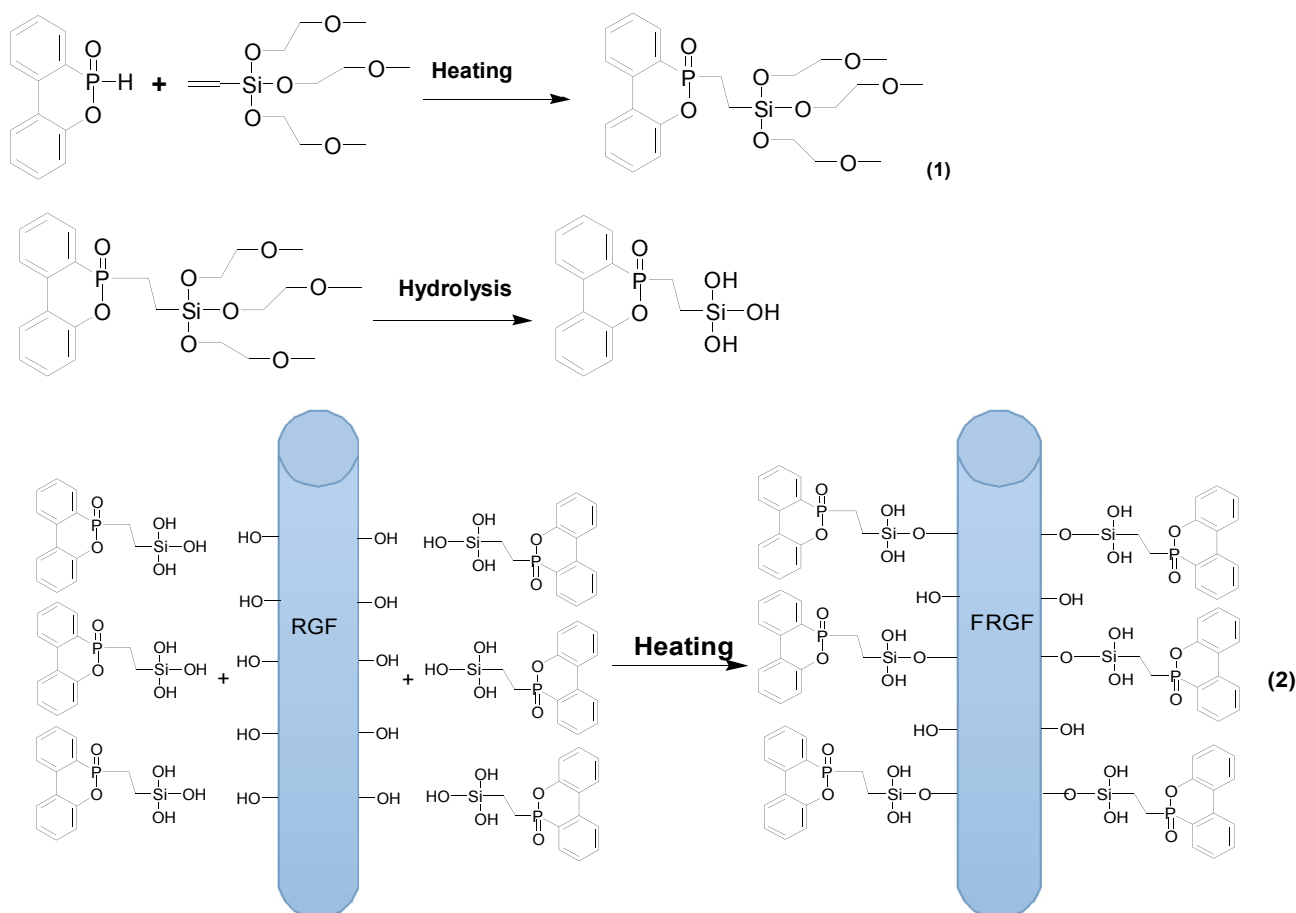
temperature was maintained at 180 °C for 4 h. After cooling to room temperature, the obtained DOPO-A172 was repeatedly washed by deionized water to remove the residual reagents, and meanwhile was hydrolyzed. The white glue was collected by filtration, and dissolved in ethyl alcohol to prepare a DOPO-A172 solution with a concentration of 30%. Glass fibers and sheets were immersed in the above solution for 20 min (the quality ratio of glass samples/solution = 100 g/800 g), and dried at 130 °C for 3 h to make glass samples be grafted with DOPO-A172. Finally, the grafted glass fibers (FRGF) and sheets (FRGS) were washed with ethyl alcohol to remove the unreacted DOPO-A172. The above synthesis can be described by Scheme 1. The graft amount of the flame retardant was calculated by the increased weight from RGF to FRGF (the calculated graft amount is 5.4 g DOPO-A172/100 g FRGF).

### 2.3. Preparation of PP resin-encapsulated glass fiber and sheets and their carbonation samples

RGF, RGS, FRGF and FRGS were coated with PP/dimethylbenzene solution (concentration: 15%), and dried in an oven at 180 °C for 2 h to remove the solvent. The obtained PP resin encapsulated-glass fibers and sheets (PP-EP-RGF, PP-EP-RGS, PP-EP-FRGF and PP-EP-FRGS) were flamed on an alcohol burner, and the coated PP resin was sufficiently burnt up or carbonized.

### 2.4. Preparation of the composites

PP pellets, GF (FRGF or RGF, its content kept a fixed 20 wt% in the entire composites) and 5.4% DOPO-A172 (only added in the bulk mode formulation) were premixed in a mixer and then the mixture



**Scheme 1.** The preparation of FRGF: (1) the reaction of DOPO and A172, and (2) the graft reaction of DOPO-A172 on GF surface.

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