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The influence of a novel two dimensional graphene-like nanomaterial on thermal stability and flammability of polystyrene



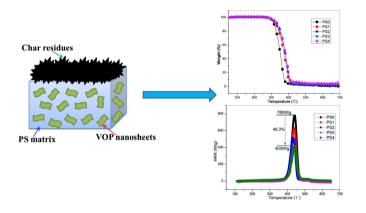


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G R A P H I C A L A B S T R A C T

A novel 2D graphene-like nanomaterial was synthesized and then modified with organic surfactants. The modified VOP nanosheets were well dispersed throughout the PS matrix, leading to remarkable improvements of thermal stability and fire safety properties.



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ABSTRACT

Significant improvements in thermal stability and fire resistance properties of polymer materials with nanofillers at low loadings hold tremendous promise for fire safety materials. In this study, a novel two dimensional (2D) graphene-like nanomaterial (VOP) was synthesized by a simple refluxing method and then modified with typical organic surfactants. Subsequently, the novel graphene-like nanomaterials were incorporated into polystyrene (PS) matrix for reducing fire hazards. The SEM and TEM images indicated that the VOP nanosheets were well dispersed throughout the matrix without obvious aggregates, leading to remarkable improvements of thermal stability and fire safety properties. With the loading of 1 wt% modified VOP, the $T_{5\%}$, $T_{10\%}$, $T_{50\%}$ and T_{max} values were increased by 15, 22, 29 and 33 °C, respectively. In addition, the presence of VOP nanosheets decreased the decomposition rate of PS and increased the amount of char residues. Furthermore, the peak heat release rate and total heat release of PS composites was decreased by 48.3% and 43.6%, respectively, compared to that of neat PS. The well dispersion, physical barrier effect and catalytic carbonization effect of VOP nanosheets were the main causations for the reduction of fire hazards.

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1. Introduction

PS is one of the most widely used thermoplastic polymers, due to its outstanding performances, mainly including heat and electric insulation, low density, excellent mechanical durability and convenience of processing and molding. However, its high intrinsic flammability accompanying with toxic gases and combustible volatiles generated during combustion is a fatal drawback, which greatly limits its further applications [1]. Therefore, it is of great significance to improve the thermal stability and flame retardancy of PS composites.

Up to now, halogenated compounds are considered to be the most effective flame retardants for PS [2,3]. However, the utilization of halogen-containing compounds usually results in releasing large amount of toxic gases and heavy smoke during combustion. Due to the increasing consciousness of environmental protection and safety concern, various kinds of halogen-free flame retardants are developed to replace halogenated ones in the flame retardant polymeric materials. Among the various halogen-free flame retardant additives, phosphorus-containing compounds are known as the effective and promising flame retardants for PS [4]. However, the incorporation of phosphorus-containing compounds usually leads to the deteriorated thermal stability of the PS composites [5]. In addition, intumescent flame retardant systems and aluminum hypophosphite are also incorporated to obtain flame-retardant PS with desired UL-94 rating and high oxygen index [6-8]. The high addition of IFR systems and aluminum hypophosphite is easy to cause deterioration in intrinsic properties of polymer matrix. Therefore, it is urgent to develop new flame retardant additive for PS that can retain or even enhance its intrinsic properties simultaneously.

Nanocomposite technology has attracted great interests in the field of flame retardant polymers due to its high efficiency and notable reinforcement. Wilkie CA et al. has reported that the addition of 3 wt% montmorillonite led to a remarkably reduction of peak heat release rate of PS nanocomposites, along with the improved mechanical strength and thermal stability [9]. Meanwhile, some other layered nanofillers such as layered double hydroxides [10], α -zirconium phosphate [11] and molybdenum disulfide [12] could also impart effective fire resistance property to PS. Similar to layered nanofillers, graphene with a unique layered structure has been incorporated into PS matrices to enhance their thermal stability and flame retardancy [13–15]. Physical barrier effect of the graphene nanosheets which slows down the diffusion of gases and degradation products, and the interface interaction between nanosheets and PS matrix, are the main reasons for these improvements [13].

Accompanying the booming development of researching on graphene, extensive attentions have been focused on the inorganic graphene-like materials including boron nitrides, carbon nitrides and transition metal dichalcogenides [16]. As an emerging 2D graphene-like material, dehydrated vanadyl phosphate (VOPO₄) has the characteristic layered structure, in which the sheets of VOPO₄ form from vertex-sharing VO₆ octahedra linking to phosphate PO₄ tetrahedra [17]. Benefiting from the inherent lamellar structure, VOPO₄ shows many attractive characteristics such as large surface area and aspect ratio, excellent catalytic activity, and the potential to delaminate within polymers. However, the VOPO₄ is mainly used as a platform for studying intercalation chemistry, the positive electrode materials for lithium batteries and supercapacitor [17–19]. As far as we know, the utilization of VOPO₄ nanosheets to fabricate polymer composite is still rare, let alone study its influence on thermal stability and fire safety properties of the polymer composites.

According to the results of published research work, nanoadditives show a better smoke and volatiles suppression effect compared to traditional flame retardants [20]. In recent years, the

catalytic flame retardancy of polymers also has attracted intensive attention. The transition metal halides and metal phosphates are usually recognized to be the catalysts for charring of polymers during thermal degradation and combustion [21–23]. Among the transition metal ions, vanadium has largely been investigated because of its redox properties which can be used to catalyze various oxidative dehydrogenations [24]. Sandro et al. has synthesized vanadium-containing saponite samples and used as potential flame retardant fillers for polymeric composites [25]. Meanwhile, phosphorus-containing compounds as halogen-free flame retardants are often used alone or combined with other flame retardants, which are able to increase the quality of charred layers. The charred layers can decrease the amount of flammable volatile gases reaching the flame zone and reduce the heat transfer from the flame to the polymer. In our previous work, the incorporation of 3.0 wt% VOPO₄ nanosheets into PVA led to obvious increments of thermal stability ((T $_{onset})$ and (T $_{50\%})$ increased by 45 $^\circ C$ and 139 °C) and fire resistance (the peak heat release rate decreased by 67%), compared to that of neat PVA [26]. Therefore, the layered VOPO₄ composed of transition metal V and phosphate is a promising nanofiller for reducing the fire hazards of polymer materials.

The aim of this work is to prepare PS nanocomposites filled with organo-modified VOPO₄ nanosheets, study the distribution of VOPO₄ nanosheets within PS, and evaluate the influence of VOPO₄ nanosheets on the thermal stability and fire safety properties of the obtained PS nanocomposites.

2. Experimental section

2.1. Materials

PS (158 K) was obtained from BASF-YPC Co., Ltd (China). Phosphoric acid (H_3PO_4 , 85 wt% solution), vanadium pentoxide (V_2O_5), 2-propanol, Cetyl trimethyl ammonium bromide (CTAB) and N, N-dimethylformamide (DMF) were purchased from Sinopharm Chemical Reagent Co., Ltd. (China). All reagents were of analytical grade and used as received without further purification.

2.2. Preparation of organo-modified VOPO₄·2H₂O

Bulk VOPO₄·2H₂O chunks were synthesized and then exfoliated to obtain ultrathin VOPO₄·2H₂O nanosheets, which had been reported in our previous work [26]. Excess amount of CTAB was dissolved in distilled water and then added to the above obtained colloidal suspension of ultrathin VOPO₄·2H₂O nanosheets under mechanical stirring for 8 h at 60 °C. The CTAB modified VOPO₄·2H₂-O which were abbreviated as C-VOP in following research was collected after centrifugation, washed with hot water, and then dried in a vacuum at 80 °C for 24 h.

2.3. Preparation of PS nanocomposites

PS nanocomposites were prepared by solvent blending method, keeping the C-VOP loading at 0.5 wt%, 1.0 wt%, 3.0 wt% and 5.0 wt %, which were named with PS1, PS2, PS3 and PS4, respectively. The pure PS was named with PS0. In a typical procedure, 10.0 g PS granules were dissolved in DMF under strong mechanical stirring to form solution A. Desired amount of modified VOP was dispersed well in DMF with ultrasonication and strong mechanical stirring to form solution B. Then the solution A and B was mixed together under ultrasonication and mechanical stirring, the obtained pale yellow slurry was dried in an oven at 100 °C for 24 h, cut into small platelets, and further dried at 130 °C in a vacuum oven for 6 h.

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