



Improving fire safety of epoxy filled with graphene hybrid incorporated with zeolitic imidazolate framework/layered double hydroxide

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ABSTRACT

In this work, a novel graphene (RGO) hybrid added with Zeolitic Imidazolate Framework/Layered Double Hydroxide (ZIF/LDH) was prepared to obtain a synergistic system (RGO-LDH/ZIF-67). Subsequently, RGO-LDH/ZIF-67 was mixed into epoxy resin (EP) by physical blending for the purpose of improving its fire safety. Based on a series of tests and analyses, it was found that RGO-LDH/ZIF-67 was beneficial to reducing the heat release of EP during its burning process. The peak heat release rate (PHRR) and total heat release (THR) of the composite with 2 wt% RGO-LDH/ZIF-67 were reduced to 464 kW m^{-2} and 37.9 MJ m^{-2} , respectively. Simultaneously, the smoke production in its flame and flameless combustion were also reduced significantly. According to the char analysis of different composites, the main mechanism is discussed. This work provided a new type of modified RGO for improving the fire safety of EP.

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1. Introduction

EP is broadly applied in the military and civil fields due to its outstanding mechanical properties, electrical insulation, chemical stability and excellent thermal properties [1–3]. However, EP is liable to combust in air, and a lot of toxic gas and smoke which endanger people's lives will be released during its combustion process, and, consequently, the application of EP is limited to some extent. In order to expand the application of EP, it is imperative to reduce its fire risk.

In recent years, more and more researchers have found that some carbon materials such as carbon nanotubes, expandable graphite and RGO can be applied for reducing the fire risk of EP [4–6]. Among those materials, RGO shows a flake structure of six-angle honeycomb lattice composed of sp^2 hybrid orbital by carbon atoms. It has received close attention because of its special two-dimensional nanostructures, and unique physical and chemical properties [7]. Recently, there have been increasing applications of RGO as an additive agent to reduce the fire hazard of polymers [8,9]. New findings indicate that RGO still has good thermal stability even when it is exposed to flame, and it shows a good flame retardant

ability for polymers, and because of the obstruction of RGO, which can effectively retard the heat transmission and volatilization of combustible gas, so as to protect the substrate, preventing the further combustion of polymers [10,11]. Nevertheless, RGO tends to agglomerate because of the existence of Vander Waals' force, which makes it difficult to be dispersed well in composites. For the purpose of surmounting this defect, much work has been done on the surface modification of RGO, and some results have shown that the agglomeration of RGO has been significantly reduced after surface modification [12,13]. Specifically, there have also been some reports about the modification of RGO with layered double hydroxides (LDHs) [14].

LDHs show good thermal stability and excellent flame retardant performance. They have gradually become a focus in the field of flame retardants. It is due mainly to the fact that LDH can provide a physical barrier effect which can prevent the evaporation of flammable gas and oxygen during the burning of EP. Furthermore, the metal oxides generated in the burning process of LDH can promote the formation of carbon residue, and make a contribution in strengthening the barrier effect. At the same time, the water vapor generated during the decomposition process of LDH will absorb heat, and reduce the temperature of composite materials' surface, thereby playing a better role in retarding the combustion of polymer [15]. In addition, there is a certain amount of positive charges

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on LDH, which facilitates the surface modification of LDH. Despite numerous reports about surface modification of LDH, there have been few reports about the modification of LDH surface by metal organic frameworks (MOFs). In view of that, this research has been undertaken.

MOFs is a kind of crystalline porous material containing interconnected forms of periodic network structure, synthesized by self-assembly reaction between inorganic metal (metal ions or metal clusters) and organic ligands. It has attracted more and more attention because of its special structure and excellent performance, especially in the field of adsorption [16–18]. However, relatively few studies have been performed on the application of MOFs as an additive agent to reduce the fire risk of polymers. There are many kinds of MOFs, such as Isoreticular metal-organic frameworks (IRMOFs), zeoliticimidazolate frameworks (ZIFs), metarial sofistitute Lavoisierframeworks (MILs) and ocket-channel frameworks (PCNs). In particular, ZIF-67, as a kind of ZIFs, is synthesized based on cobalt ions and 2-methylimidazole, containing flame retardant elements of Co and N, and the metal oxide (Co_3O_4) generated during its combustion process has a catalytic effect which facilitates the generation of a dense char layer to prevent the substrate from further burning, and thus to accomplish the aim of reducing fire risk. Therefore, ZIF-67 was selected in this work to further reduce the fire hazard of polymer.

To our knowledge, a flame retardant with a single component can decrease the fire risk of composite materials when the amount of additive is large, but its flame retardant efficiency is rather low. However, a flame retardant containing more than one component would form a synergetic system, so as to better reduce the fire hazard [19,20]. Therefore, in this study, dual modification of RGO (RGO-LDH/ZIF-67) was performed by connecting RGO, LDH and ZIF-67 (as shown in Scheme 1). The morphology of the prepared flame retardants was studied. Subsequently, RGO-LDH/ZIF-67 was added into EP through physical blending. The effects of RGO-LDH/ZIF-67 on reducing the fire risk of EP were studied, and their main mechanism was also investigated.

2. Experimental

2.1. Materials

H_2SO_4 (98%), Graphite powder (spectral purity), NaNO_3 , KMnO_4 , HCl (37%), H_2O_2 (30%), H_2N_2 (80%), NaOH , ethyl alcohol, cobalt nitrate, 2-methylimidazole, absolute methanol, magnesium nitrate, aluminum nitrate, sodium carbonate anhydrous were bought from Shanghai Jutai Special Reagent Co., Ltd. (China). Epoxy resin (E-44) was bought from Wuxi Borui Chemical Technology Co., Ltd. (China). 3,3'-Dichloro-4,4'-diaminodiphenylmethane (MOCA) was bought from Shandong Guangxun Chemical Co., Ltd. (China).

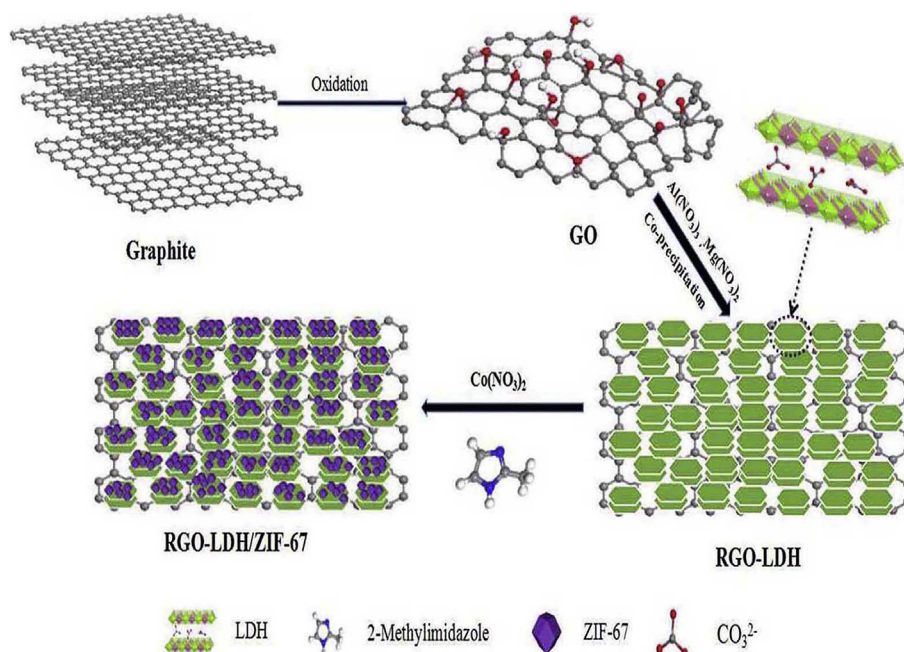
2.2. Fabrication of flame retardants

2.2.1. Fabrication of RGO-LDH

GO was made with modified Hummer's method [21]. RGO-LDH was fabricated with a simple coprecipitation method as follows: Firstly, 0.32 g GO was dissolved in 100 ml deionized water, with addition of some sodium hydroxide and anhydrous sodium carbonate, and ultrasonic reaction for 1.5 h; the obtained homogeneous solution was poured into a 500 ml flask. Subsequently, magnesium nitrate (1.28 g) and aluminum nitrate (1.88 g) were dissolved completely in 200 ml water to obtain a uniform system, and this uniform liquor was added into the dispersion of GO by dropping. This system was adjusted to a PH of 10, and stirred for 24 h at 60 °C. Then hydrazine hydrate solution (0.75 ml) was added into the above system and its temperature was increased to 100 °C. The system was cooled down to room temperature after stirring for 2 h, washed repeatedly, and freeze dried to obtain RGO-LDH. RGO and MgAl-LDH were also obtained in the same way.

2.2.2. Fabrication of RGO-LDH/ZIF-67

RGO-LDH/ZIF-67 was fabricated with a simple process in the following steps: RGO-LDH (1 g) was ultrasonically dispersed in an appropriate amount of deionized water to acquire a homogeneous system (A), and 6.56 g 2-methylimidazole was dissolved in 100 ml



Scheme 1. Illustration of ZIF-67 modification of RGO-LDH.

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