



Curing characteristics of an epoxy resin in the presence of functional graphite oxide with amine-rich surface



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ARTICLE INFO

Article history:

Received 15 March 2015

Received in revised form 1 June 2015

Accepted 3 June 2015

Available online 11 June 2015

Keywords:

Epoxy resin

Cure kinetics

Functional graphite oxide

Amine-rich surface

Curing reaction pathways

ABSTRACT

Functional graphite oxide (DGO) with amine-rich surface was successfully prepared through the amidation reaction and characterized by X-ray diffraction analyses (XRD), Fourier transform infrared spectra (FTIR) and Raman spectra. The effects of functional graphite oxide on the curing kinetics of epoxy (EP) were investigated by means of differential scanning calorimetry (DSC). The curing kinetic parameters of EP, EP/graphite oxide (GO) and EP/functional graphite oxide were obtained. There was not much difference in total heat of reaction ΔH and peak temperature T_p with the incorporation of GO or DGO. However, the activation energy, E_a , and the overall order of reaction $m+n$ were enhanced. Comprehensive kinetic analyses indicated that the incorporation of GO sheets brought in an effect of inhibition on curing process. While the inhibition effect weakened when the GO is modified with amine-rich surface. The possible curing mechanism and reaction pathways were proposed to provide a reasonable explanation.

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1. Introduction

Epoxy resins are high-performance thermosetting polymers that display a unique combination of properties such as low cure shrinkage, compatibility with a great number of materials, high mechanical strength, adhesion, chemical resistance and good electrical insulation [1]. However, epoxy resins are brittle and have poor resistance to the crack propagation. In general, the properties of epoxy resins are always determined by the chemical structure of the resin itself, the hardener and the fillers added, as well as the cross-linked network achieved after curing [2]. In order to make up the weak point of the epoxy resins and get more excellent properties, various nano-particles are used to toughen the epoxy resins [3]. In order to achieve good mechanical properties, thermosetting materials are generally cured with high crosslink density. However, it always brings in poor fracture resistance. In general, effective toughening of thermosetting materials means an improvement of fracture properties and simultaneously does not influence the mechanical properties of the matrix [4]. Very recently, the enhancement of toughness on thermosetting materials by the incorporation of graphene has been studied [5].

Graphene, composed of single-atom-thick sheet of hexagonally arrayed sp^2 -bonded carbon atoms, has been under the spotlight owing to its intriguing and unparalleled physical properties [6]. Researchers working upon this exciting material, have reported graphene to possess remarkable mechanical, electrical and thermal properties [7–10]. It is now recognized as the strongest material in the world, with a Young's modulus of 1 TPa and breaking strength of 130 GPa, according to the research of Lee et al. [11]. Meanwhile, high tendency of pristine graphite to agglomerate necessitates chemical oxidation of graphite to assist in proper dispersion of graphene in polymer matrix [12]. Oxidation of graphite leads to oxygen containing groups' attachment (such as epoxide, hydroxyl and carboxyl groups) on graphite surface which can alter the van der Waals interactions significantly. Meanwhile, the presence of functional groups supports the intercalation of macromolecules into the interlayer spacing of graphite sheet [13,14]. However, graphite oxide can only be dispersed in aqueous media, which is incompatible with most organic polymers. Therefore, graphite oxide followed by chemical functionalization has become a particularly attractive target because it can improve the solubility and processability as well as enhance the interactions with organic polymers [15,16]. Considerable work has been carried out on this, including the reduction of graphite oxide (GO) in a stabilization medium [17], covalent modification by the amidation of the carboxylic groups [15,16], non-covalent functionalization of

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reduced graphene oxide [18–20], nucleophilic substitution to epoxy groups [21], and diazonium salt coupling [22].

Generally, improvement in the properties of epoxy nanocomposites depends on the formation of the crosslinked molecular network, which is often influenced by the mechanism and kinetics of epoxy resin curing that involves various chemical reactions [23]. In this sense, an understanding of curing performance of the nano-modified epoxy matrix can hence contribute to an improved design and manufacturing for the resulting composite parts. In past decades, extensive studies concerning mechanisms and kinetics of epoxy resin cure have been presented in the literature [24–26]. Some studies have reported the curing performance and kinetics of epoxy nanocomposites filled with graphene oxide (GO) [4,27]. However, few studies have been published to depict the curing mechanism and kinetics of epoxy nanocomposites with the existence of functional graphite oxide (DGO).

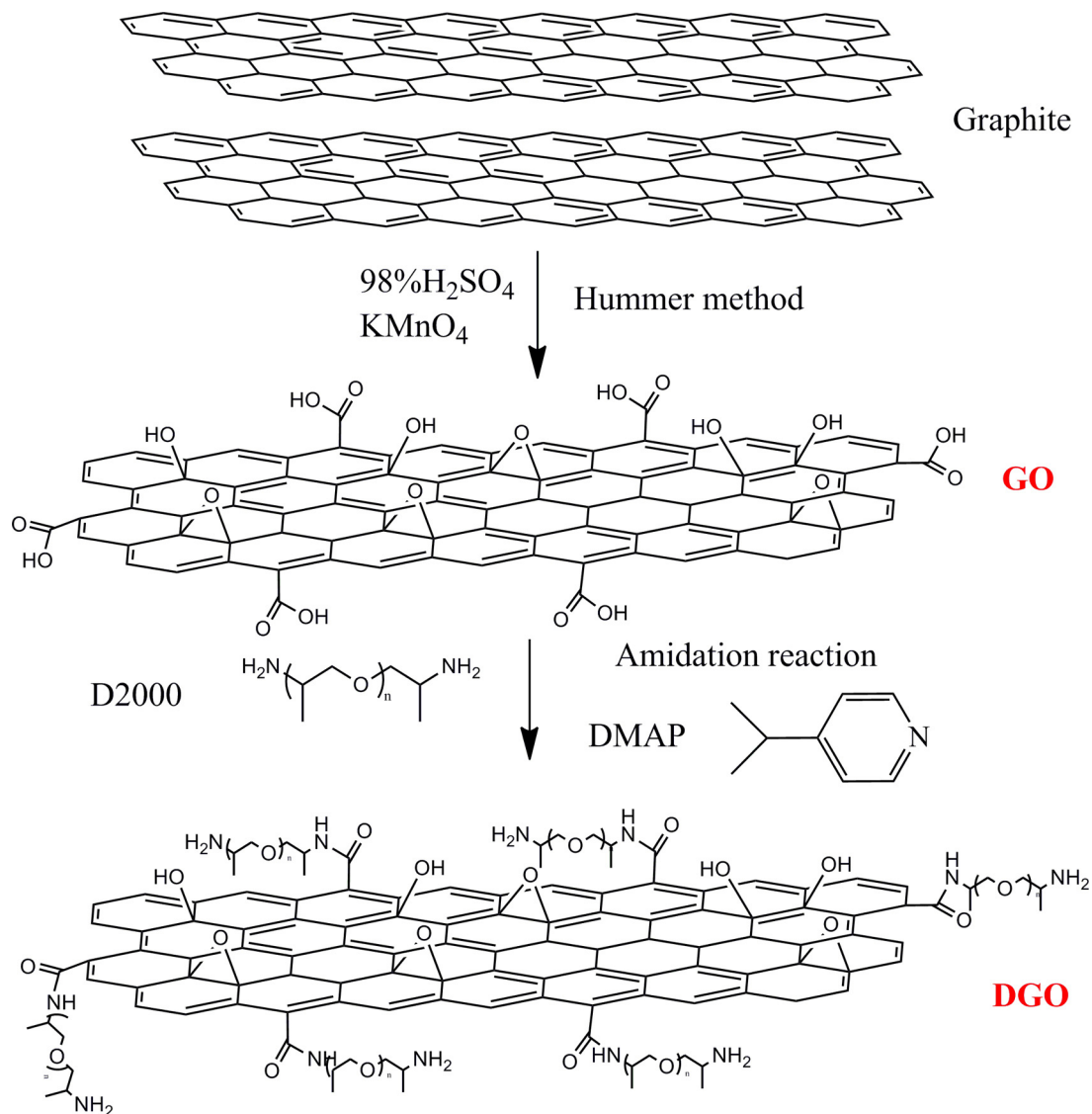
In our study, we successfully prepared functional graphite oxide (DGO) with amine-rich surface. In detail, graphite oxide was covalently modified through the amidation reaction between carboxylic groups and poly(oxypropylene)diamine. Compared with neat epoxy system and epoxy/GO system, the aim of present work was to study the incorporation of functional graphite oxide

(DGO) with amine-rich surface on the curing behavior and mechanism of epoxy resin. For this propose, non-isothermal DSC measurements was carried out to reveal the curing behavior and some empirical approaches were used to model the kinetics of the curing reaction.

2. Experimental

2.1. Materials

The diglycidyl ether of bisphenol A type (E44) epoxy resin was provided by Deyuan Chemistry Plant, China, with an epoxide equivalent weight of 210–240 g/equiv. The density is 1.18 g/cm³ at 25 °C and the viscosity is 2500 mPa s at 40 °C. The curing agent was 4,4-diamino diphenyl methane (DDM), which was provided in analytical grade by Shanghai crystal neat reagent Co. (Aladdin Reagent Co., China). The natural graphite was provided by Qingdao Dongkai Graphite Co., Ltd. (Qingdao, China). The graphite has a particle size of 45 μm with a density of 2.25 g/cm³. Poly(oxypropylene)diamine were obtained from Huntsman Corporation (The Woodlands, TX, USA), with average weight molecular weight of 2000 g/mol (D2000). Sodium nitrate (NaNO₃), potassium



Scheme 1. Reaction scheme of grafting poly(oxypropylene)diamine onto GO surface.

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