

Enhanced thermal stability and structural characteristics of different MMT-Clay/epoxy-nanocomposite materials

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Abstract

Epoxy–clay nanocomposites, HDTMA–BDGE, HDTMA–BPDG, HDTMA–BBDG, HDTMA–TGDDM and HDTTPP–BDGE were synthesized using hexadecylammonium clay and hexadecylphosphonium clay, respectively. The Montmorillonite (MMT) clay was modified with quaternary ammonium salt and with triphenylphosphonium salt which was intercalated into the interlayer region of MMT-Clay. The epoxy–clay systems were cured by using diaminodiphenylsulphone as a curing agent. The X-ray diffraction patterns obtained for the systems confirmed the nanodispersion of MMT-Clay in the epoxy networks. The ammonium clay-modified systems displayed appreciable mechanical and glass-transition temperature properties while, the phosphonium clay-modified system exhibited highest thermal resistance properties compared with unmodified epoxy systems. The T_g decrease observed in all the clay-modified epoxy systems, may be compromised with their advantage of requiring the filler content very low (5wt%), when compared to the conventional epoxy systems whose filler quantity is normally required from 25 to 30 wt%.

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1. Introduction

Epoxy resins generally possess a combination of properties that makes it particularly suitable for use as structural components. However, epoxies suffer from being brittle or lack of enough strength, and therefore to remedy this, they may be blended with fillers to improve its impact strength. Although the added fillers may improve the impact strength of plastic articles, but there is a concomitant decrease in tensile and flexural strength and modulus of the modified epoxies. Thus, there is a need for an appropriate selection of fillers that provide optimum impact strength while minimizing the reduction in strength and modulus. One way to improve the mechanical and melt processing properties of epoxy resin is by adding MMT clay particles. The clay particulates were added as

reinforcement fillers in the preparation of polymer–clay nanocomposites. These clay particulates were dispersed in the form of nanosized clay platelets having around 1–3 nm thickness and a few hundred nanometers length. The high aspect ratio of the clay and the occupation of large volume fraction of the clay in the reinforcement of the composites are expected to show better mechanical, physical and thermal properties over conventional composites.

Several researchers have undertaken the study of the clay dispersed epoxy polymer composite materials. Kornmann et al. [1] have reported the modified MMT clay incorporated epoxy resins cured with different amines. Shin et al. [2] have studied clay modification in poly-dimethylsiloxane containing isocyanate grouped-epoxy resins. Becker et al. [3] reported three types epoxies loaded with octadecylammonium modified MMT-Clay and cured using diethyltoluenediamine isomers. The effects of the nanofillers in polymer composites on the thermal behaviour of the different filler-resin composites have been studied [4–6]. The increase and as well the decrease

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in T_g temperature, that are dependent on the nanofiller quantity added to the epoxies have been reported [7]. Messersmith and Giannelis [8] have analyzed the effect of different curing agents and curing conditions in the formation of nanocomposites from bisphenol A diglycidyl ether and organo-MMT [bis (2-hydroxyethyl) methyl hydrogenated tallow alkylammonium cation]. They found that the modified clay disperses readily in bisphenol A diglycidyl ether and the intercalation improved if the temperature raised from room temperature to 90 °C. So far the reported literature was on the epoxy–clay nanocomposites based on bisphenol A diglycidyl ether [9–15]. However, there are very few reports on the systematic study on high performance graded epoxies that were especially loaded with the high temperature resistant moiety functionalized nanoclays such as hexadecyltrimethylammonium clay and hexadecyltriphenylphosphonium clay to improve the thermal and mechanical properties of the materials [16].

Therefore, the motivation of our work here is to prepare high performance epoxy–clay nanocomposites from the different epoxy resins blended with two high temperature resistant organo-MMT-Clays dispersed using 5wt% and systematically investigate the property changes with a view to understand the effect of polymer–clay in terms of their epoxy–nanoclay miscibility, processing stability, thermal stability and toughening properties. In this study, we have used different epoxies such as, bisphenol A diglycidyl ether (BDGE), bisphenol A propoxylate diglycidyl ether (BPDG), bisphenol A brominated diglycidyl ether (BBDG) and tetraglycidyl of diaminodiphenylmethane (TGDDM) to synthesize high performance nanocomposites. Curing of these epoxide monomers with diaminodiphenylsulphone (DDS) and nanosized MMT-Clay has yielded three dimensional cured materials that combined with excellent thermal stability, and high mechanical strength properties.

2. Experimental

2.1. Materials

Bisphenol A diglycidyl ether (182 Epoxy equivalent weight, LY556 Araldite, Ciba-Geigy), bisphenol A propoxylate diglycidyl ether (PO/phenol 1, Aldrich), bisphenol A brominated diglycidyl ether (mol. wt. 350–450 g/epoxide, Aldrich), and tetraglycidyl of diaminodiphenylmethane synthesized [17] were used as the polymer matrix. Diaminodiphenylsulphone (m.p. 174–178 °C, Fluka) solid was used as curing agent in the systems. Montmorillonite layered silicate clay with an initial interlayer d -spacing of 10.09 Å was provided by the Aldrich chemical company. Hexadecyltrimethylammonium-bromide, hexadecylbromide and triphenylphosphine (Aldrich) were used to chemically modify the clay surfaces.

2.2. Preparation of hexadecyltrimethylammonium (HDTMA)–MMT organoclay

The HDTMA–MMT organoclay was synthesized by the ion exchange reaction between Na^+ MMT and

hexadecyltrimethylammoniumbromide (HDTMABr) [6]. A 6.5 g of Na^+ MMT was suspended in 400 ml of distilled water and stirred for 2 h. The suspension was heated to 60 °C and an aqueous solution of 21 mmol of HDTMABr salt was added gradually under stirring for 1 h. Thus the obtained organoclay was centrifuged and washed with distilled water until no bromide ion was estimated using 0.1 N AgNO_3 solution. The organoclay was then dried in a vacuum oven at room temperature and powdered using a mortar and pestle.

2.3. Preparation of hexadecyltriphenylphosphonium (HDTTPP)–MMT organoclay

The HDTTPP–MMT organoclay used was synthesized by ion exchange reaction between Na^+ MMT and hexadecyltriphenylphosphoniumbromide (HDTTPPBr). In a 500 ml beaker, 6.1 g (0.02 mol) of bromohexadecane, 15.73 g (0.06 mol) of triphenylphosphine and 300 ml of tetrahydrofuran were placed and the solution was heated at 80 °C for 24 h. After, 24 h the suspension was allowed to settle and the solvent was decanted. The precipitated HDTTPPBr was filtered and dried. The obtained HDTTPPBr (11.34 g, 0.02 mol) was dispersed in a solution containing 300 ml of deionized water, 75 ml of ethanol and 7.5 ml of conc. Hydrochloric acid at 80 °C. The HDTTPPBr solution was added with a solution of 15 g of Na^+ MMT in a 300 ml of deionized water and stirred vigorously for 3 h. The obtained HDTTPP–MMT organoclay was then dried in a vacuum oven at room temperature and powdered using a mortar and pestle.

2.4. Preparation of organoclay/epoxy-nanocomposites

Epoxy resin was preheated at 90 °C to lower the viscosity and make it easier to disperse the added clay. The 5wt% organoclay was added to the 100 parts of epoxy/DDS system using mechanical shear mixer at 1000 rpm for 2 h. The blending durations were varied according to the viscosity of the system in order to effectively disperse the clay. The high speed mixing assisted the clay to break into small particles and thereby helped to increase the clay–epoxy interfaces. After thorough mixing up of organoclay and the resin, DDS curing agent was added according to their stoichiometric ratio. The mixture was heated at 140 °C for 5–10 min to react the DDS with the epoxy to obtain a liquid prepolymer with a partial conversion which became completely transparent and homogeneous. The obtained prepolymer was poured into a stainless steel mold that was preheated at 140 °C, cured at 140 °C for 3 h, 160 °C for 2 h and followed by a post-curing at 180 °C for 4 h. The nanocomposite synthesized was characterized using several techniques.

2.5. Characterization

The FTIR spectra of the organoclays dispersed in potassium bromide discs were recorded with Nicolet (model 20DXB) spectrophotometer, scanning range 400–4000 cm^{-1} . X-ray diffraction studies were performed to evaluate the degree of

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