



Jatropha curcas oil based alkyd/epoxy/graphene oxide (GO) bionanocomposites: Effect of GO on curing, mechanical and thermal properties



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ABSTRACT

Jatropha curcas oil based alkyd/epoxy/GO bionanocomposites were prepared by direct solution blending of alkyd/epoxy blend matrix with GO nano filler. Structures and properties of the bionanocomposites were characterized with Fourier transform infrared spectroscopy, X-ray diffraction, transmission electron microscopy, differential scanning calorimetry, thermogravimetric analysis, and tensile testing. X-ray diffraction and transmission electron microscopy study demonstrates the formation of highly exfoliated GO layers and its homogeneous dispersion throughout the polymer matrix with 1 and 3 wt% GO. However, the intercalated structure is predominant with 5 wt% GO. The homogeneous dispersion and the strong interaction of the GO layers and the polymer matrix induced the significant improvement in thermal and mechanical properties of the bionanocomposites. The tensile strength and elastic modulus of the bionanocomposite increased by 133% and 68% respectively with 3 wt% GO loading. The thermal stability of the bionanocomposite improved by 39 °C and T_g is shifted toward higher temperature by 20 °C as compared to the pristine polymer. Incorporation of GO significantly decreases the curing time of the alkyd/epoxy resin blend.

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1. Introduction

During recent years, increased attention has been devoted to the development of bionanocomposites. This effort is justified both by the potential technological applications of these materials and by the environmental issues which they resolve [1]. Bionanocomposites are a class of materials consisting of a natural polymeric matrix reinforced with organic/inorganic filler having at least one dimension on the nanometer scale. The bionanocomposite shows the significant advantages of exhibiting biodegradability and biocompatibility in various applications, agricultural, medicinal, drug release, packaging fields [2,3].

Vegetable oil based alkyd resins have a number of advantages, including versatility in structure and properties, overall low cost, ease of application and are renewable. However, they suffer some major drawbacks such as poor alkali resistance, inferior mechanical properties, low hardness and thermal stability and long curing time as compared to epoxy resins. To improve these drawbacks,

alkyd resins are blended with other suitable resins, such as epoxy resin, amino resin, silicone resin and ketonic resin. The thermal and mechanical properties of these polymers can be improved further by the decoration of nanocomposites [4,5].

Nanocomposites containing multifunctional nano fillers such as graphite, carbon nanotubes (CNT), fullerenes, and silicates are being developed with good mechanical, thermal, and electrical properties [6–8]. In our earlier work we have reported the preparation of *Jatropha curcas* oil based alkyd/epoxy/EG biocomposite with significant improvement in thermal and mechanical properties [5]. Yasmin et al. studied the structural, mechanical, viscoelastic and thermal properties of graphite platelet/epoxy nanocomposite. The nanocomposite showed good thermal, mechanical, and rheological behavior [9]. Bharadwaj et al. reported the preparation of unsaturated polyester/organically modified clay nanocomposites, cross-linked with methyl ethyl ketone peroxide (MEKP) with good thermal, mechanical, and rheological properties [10]. Seyhan et al. prepared CNT/polyester nanocomposites using three-roll mill and sonication techniques. The CNT/polyester blend exhibited a shear thinning behavior, while polyester matrix acts as a Newtonian fluid. Nanotubes modified with amine functional groups showed superior tensile strength, as compared to those with untreated

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CNTs [11]. Battisti et al. investigated the electrical conductivity of unsaturated polyester/multiwalled CNTs nanocomposites. The nanocomposite found to be more conducting with percolation threshold at 0.026 wt% CNTs [12].

During the last half decade, GO, a two-dimensional single sheet of graphite oxide has attracted a great deal of interest. It is because of its low cost, unique structure, remarkable properties. GO can be prepared in large scale from low cost natural graphite which is an easily available material [13–15]. The presence of oxygen functional groups such as hydroxyl, carboxyl, carbonyl and epoxide facilitate easy dispersion of GO sheets in polar solvents. Moreover, the functional groups in the GO sheets impart strong interaction with polar molecules or polymers to form GO-intercalated or exfoliated composites [16–18]. It offers the possibility of making multifunctional nanocomposites in a cost effective way over other expensive fillers like CNT. There are several reports on GO based polymer nanocomposites with significant improvement in the thermal, mechanical and electrical properties. Wang et al. studied the curing dynamics and network formation of cyanate ester resin/GO nanocomposites. The incorporation of GO into the resin matrix showed a strong catalytic effect on the cure of the resin, and addition of 4 wt% GO resulted in the decrease of curing temperature to 97 °C [19–21].

Inspiring from those studies, the objective of our present work is to fabricate bionanocomposite films that contain reinforcing GO nanofillers in an alkyd/epoxy blend matrix and to evaluate mechanical and thermal properties. The structure and property relationship of the bionanocomposites are also investigated. This study aims at obtaining a well dispersion of GO sheets throughout the polymer matrix as well as to accomplish significant improvement in thermal and mechanical properties. The introduction of minute amount of GO greatly enhance the properties of alkyd/epoxy blend matrix.

2. Experimental

2.1. Materials

J. curcas oil based alkyd resins were synthesized as we have reported earlier [22]. Epoxy resin (Epoxy equivalent weight: 170–180 g/eqv.) and hardener poly(amido amine), MEKP and Co-octoate of commercial grade (Kumud Enterprises, Kharagpur, West Bengal, India) were used as received. Graphite flakes was purchased from Sigma–Aldrich with a particle size of 150 μm and purity of 99.9%. All of the other solvents and materials were of analytical grade, commercially available and used without further purification.

2.2. Preparation of GO

GO prepared from natural graphite using a modified Hummers method [23]. In a typical method graphite (5 g) and NaNO₃ (2.5 g) were mixed with H₂SO₄ (120 mL) in a 500 mL beaker and the mixture was stirred for 30 min in an ice bath. Under vigorous stirring, 15 g of KMnO₄ was added to the suspension at a controlled rate to keep the reaction temperature below 20 °C. The mixture was allowed to stir for 12 h at room temperature. Afterward 150 mL of distilled water was added slowly to the reaction mixture and allowed to stay for 24 h under vigorous stirring. Then 50 mL 30% H₂O₂ was added and stirred for 6 h. Finally the mixture was washed with 5% of HCl followed by water until the pH of the filtrate was 7 and dried in a vacuum oven.

2.3. Preparation of alkyd/epoxy/GO bionanocomposite

The alkyd/epoxy/GO bionanocomposite was prepared by solution blending method. The alkyd and epoxy was blended at the

Table 1
Compositions of the bionanocomposites.

| Entry | Sample particulars | Alkyd (g) | Epoxy (g) | GO (wt%) |
|-------|--------------------|-----------|-----------|----------|
| 1 | BNC0 | 2 | 2 | 0 |
| 2 | BNC1 | 2 | 2 | 1 |
| 3 | BNC3 | 2 | 2 | 3 |
| 4 | BNC5 | 2 | 2 | 5 |

ratio of 1:1 in acetone (1 mg/mL) and then GO was added in different wt% (Table 1). The mixture was stirred by mechanical stirrer followed by ultra-sonication for 1.5 h to get exfoliated and homogeneous dispersion of the GO nano-sheets in the polymer matrix. The solvent was evaporated at 55 °C and the mixture was dried at 40 °C in a vacuum oven until it was completely bubble free. Afterward, poly (amido amine) (50 wt% with respect to the epoxy resin) along with MEKP and Co-octoate (4 and 2 wt% respectively with respect to the alkyd resin) were added to the mixture. The mixture was then placed on a teflon sheet by an applicator maintaining the film thickness of 0.3 mm and dried under vacuum in a desiccator for overnight at ambient temperature. The bionanocomposite was allowed to cure at 70 °C for further study. The curing time of the bionanocomposites was recorded.

2.4. Characterization

The viscosity of the composites under a constant shear stress of 100 Pa is measured by a modular compact rheometer (MCR 500, Physica, Anton Paar) at 25 °C. Fourier-transform infrared (FTIR) spectra were recorded on a Nicolet, Impact 410 FTIR spectrometer at room temperature over a frequency range of 4000–500 cm⁻¹. X-ray diffraction (XRD) patterns were obtained using a Rigaku X-ray diffractometer (Miniflex, UK) using Cu Kα (λ = 0.154 nm) radiation at a scanning rate of 2° min⁻¹ with an angle ranging from 2° to 60°. To study the thermal degradation of the bionanocomposites, thermogravimetric analysis (TGA) was carried out on a Shimadzu TGA 50, thermal analyzer in nitrogen atmosphere at a heating rate of 10 °C min⁻¹ in the temperature range 25–600 °C. The glass-transition and crystallization behaviors were investigated by differential scanning calorimetry using a Shimadzu DSC-60 in nitrogen atmosphere. The analysis was run at a scanning speed of 10 °C min⁻¹ from 25 °C to 250 °C. The surface morphology of the specimens was studied by scanning electron microscope (SEM) of model JSM-6390LV, JEOL, Japan at an accelerating voltage of 5–15 kV. The surface of the specimens was coated with platinum before the SEM analysis. To study the dispersion and delamination of GO sheets, a PHILIPS CM 200 transmission electron microscope (TEM) was used (200 kV). The tensile strength, elongation, and elastic modulus of the bionanocomposites were measured on a universal tensile testing machine (Zwick Z010, Germany) at ambient conditions. The extension rate was 5 mm/min and the load cell was 10-kN, with a gauge length of 40 mm. The specimen dimension was 60 mm in length, 10 mm in width, and 0.3 mm in thickness. Three parallel measurements were carried out for each sample.

3. Results and discussion

Alkyd/epoxy/GO bionanocomposite films were prepared by dispersing different concentrations of GO in alkyd/epoxy resin blend matrix and subsequently cross-linked. The bionanocomposite film is highly flexible and shown in Fig. 1.

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