

# Expanded graphite as a filler for epoxy matrix composites to improve their thermal, mechanical and electrical properties

Subhra Gantayat<sup>1,2</sup>, Gyanaranjan Prusty<sup>1</sup>, Dibya Ranjan Rout<sup>2</sup>, Sarat K Swain<sup>1,\*</sup>

<sup>1</sup>Department of Chemistry, Veer SurendraSai University of Technology, Burla, Sambalpur-768018, India

<sup>2</sup>School of Applied Science (Physics), KIIT University, Bhubaneswar-751024, India;

**Abstract:** Expanded graphite (EG)-reinforced epoxy composites were prepared by a solution mixing method. The structure and morphology of the EG/epoxy composites were investigated by XRD, FE-SEM and HR-TEM. The EG prepared by acid oxidation and thermal expansion shows good compatibility with the epoxy resin that enters the EG layers to decrease their thickness to 60-70 nm, owing to its abundant oxygen-containing functional groups. With the addition of 9 wt% EG, the thermal decomposition temperature of the composite increases from 340 to 480 °C, the electrical conductivity from  $10^{-15}$  to  $10^{-5}$  S/cm and the tensile stress is increased by more than 30%. These improvements are attributed to the good dispersion of EG sheets in the epoxy matrix.

**Key Words:** Expanded graphite; FE-SEM; HR-TEM; Conductivity

## 1 Introduction

Polymer matrix composites are multi-phase materials produced by combining polymer resins with reinforcing fillers having improved properties in comparison with the matrix materials. Hence, different fillers are used to enhance the physical and mechanical properties of composites. Polymer matrix composites are of scientific and industrial interest because of their enhanced properties arising from the reinforcing function of fillers<sup>[1-4]</sup>. Different conducting fillers such as carbon nanotubes and graphite have been extensively studied because of their ability to increase the mechanical, thermal and electrical properties of the native polymers<sup>[5,6]</sup>.

Epoxy resins are a class of thermoset materials available in various forms from low viscosity liquid to high melting solids, which are widely used as polymer matrices in composites, owing to their high strength, low shrinkage, excellent adhesion to substrates, chemical resistance and low cost. Most of polymers are generally electrical insulators with very low concentrations of free charge carriers. Thus they are non-conductive and transparent to electromagnetic radiations. This property made them incapable for the use as enclosures for electronic equipments. Hence, these limitations are the causes of growing research activities for electrically conducting polymers. Conducting polymers can be either inherently conductive or insulating polymers composited with

conductive fillers. Conductive composites are used in light emitting devices, batteries, electromagnetic shielding and other functional applications<sup>[7-9]</sup>. Conductive fillers such as carbon black, carbon nanotubes and graphite have been extensively investigated<sup>[10-16]</sup>. These fillers effectively improve the electrical conductivity of the polymers. The significant increase in electrical conductivity with the filler content has been observed for most composites, which could be explained by the percolation transition from the formation of the conductive network<sup>[17]</sup>.

In comparison to carbon nanotubes, graphite continues to attract considerable attentions because of their mechanical and electrical properties, low density, easy processing and low cost. Graphite exists as a layered material and the layers are packed closely by Van der Waals' force. For an efficient utilization of graphite as filler in a polymer composite, its layers must be partly separated to obtain expanded graphite that is dispersed throughout the polymeric matrix. Also in its natural form, little reactive groups exist on the graphite and as a result, it is difficult to intercalate monomers into the graphite interlayer to form a composite. If the raw graphite is used as reinforcement, it is not possible to disperse graphite layers in epoxy matrix. The EG is prepared when raw graphite is exposed to strong oxidizers such as nitric acid (HNO<sub>3</sub>), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) or potassium permanganate (KMnO<sub>4</sub>). In comparison to raw graphite, the EG sheets are heavily

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\*Corresponding author. E-mail: swainsk2@yahoo.co.in

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oxygenated having hydroxyl and epoxide functional groups on their basal planes, in addition to carbonyl and carboxyl groups located at the sheet edges. The presence of these functional groups makes them strongly hydrophilic. EG can be readily dispersed in water and incorporated into polymer matrices with a help of these functional groups for the preparation of composites. Chen et al. [18] measured the tensile strength of the EG/polystyrene composite and found that its tensile strength is a little higher than that of the pure polystyrene. Kim et al. [19] compared the thermal property of virgin polylactic acid with that of the EG/polylactic acid composites, and found that the thermal stability of the composites increased with the EG content. Xiao et al. [20] measured the thermal property of the polystyrene/graphite composite and reported a thermal degradation temperature of the composite 20 °C higher than that of pure polystyrene.

Though graphite was extensively investigated as filler in polymer matrix composites, EG was paid less attention. In the present study, the dispersion of EG in epoxy matrix to prepare EG/epoxy composites was investigated to reveal its influence on the mechanical, thermal and electrical properties of EG/epoxy composites.

## 2 Experimental

### 2.1 Materials

Epoxy resin was purchased from Merck, India. Concentrated  $H_2SO_4$  and  $HNO_3$  were analytical grade chemicals and used directly without any further purification. Graphite fine powder with an average diameter of 500  $\mu m$  was purchased from Loba chemical Pvt. Ltd., India for preparing the EG.

### 2.2 Preparation of EG

Raw graphite was first dried in a vacuum oven for 24 hours at 100 °C. Then a mixture of concentrated  $H_2SO_4$  and  $HNO_3$  with a volume ratio of 4:1 was added slowly to a glass flask containing graphite powder with vigorous stirring. After 24 h of reaction, the acid treated graphite powder was filtered and washed with deionised water until the pH value of the filtrate reached 6.4. After drying at 100 °C for 24 h, the resulting graphite intercalation compound was subjected to a thermal shock at 900 °C for one minute in a furnace to form the EG.

### 2.3 Synthesis of EG/ epoxy composites

EG/epoxy composites were synthesized to have different contents of EG (3, 6, and 9wt% based on epoxy weight) by a solution mixing method. Calculated amount of epoxy and EG were separately dispersed in deionised water at ambient temperature via stirring for half an hour. The EG suspension was added to the epoxy solution and stirring was continued for 3 h. The resulting solution was centrifuged for 15 min and the

resulting sample was dried in an oven at 50 °C. The detail synthetic process is illustrated in Fig. 1.

## 2.4 Characterization

X-ray diffraction (XRD) of the composites was carried out by a Rigaku X-ray diffractometer (Model No. P.DD966) with  $CuK\alpha$  radiation at 40 kV and 150 mA. The morphology and dispersion of the EG in epoxy resin were investigated by using a field emission scanning electron microscope (JEOL-JSM-5800). A high resolution transmission electron microscope (Tec-nai 12, Philips) operating at 120 kV was used to study the dispersion of EG in epoxy matrix. Mechanical properties of the EG/epoxy composites were measured with ASTM-D-638-00 using an Instron testing machine (Model-5567) and the test was performed at a rate of 50 mm/min with a load of 0.5 ton. The five specimens for each composition were used for measurement and average values are reported. The TGA analysis was carried out by taking the sample in the pan (8-10 mg) and the temperature was increased by 10 °C per minute and heated up to 800 °C. Conducting measurement was carried out using LCR-Hi Tester, HOIKI after the sample being processed into pellet form.

## 3 Results and discussion

### 3.1 Structural analysis

The XRD patterns of raw graphite (RG), epoxy and the EG/epoxy composites are shown in Fig. 2.

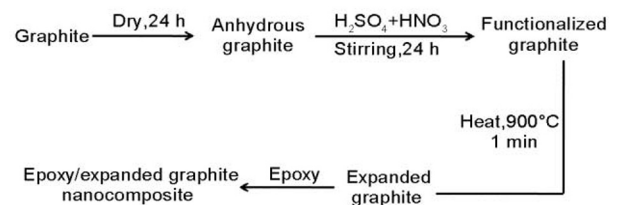


Fig. 1 Schematic representation for the preparation of EG/epoxy composite.

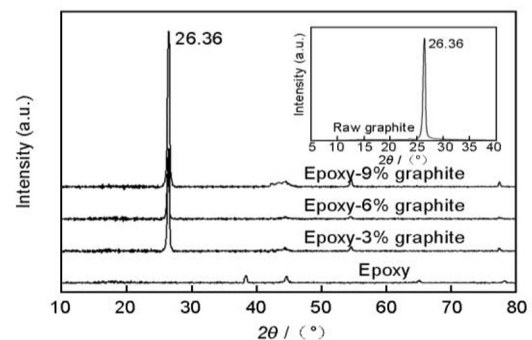


Fig. 2 XRD pattern of Epoxy and EG/epoxy composite at different percentage of EG concentration and XRD of raw graphite (Inset).

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