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Thermo-mechanical characterization of siliconized E-glass fiber/hematite particles reinforced epoxy resin hybrid composite

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ABSTRACT

In this present work hybrid polymer (epoxy) matrix composite has been strengthened with surface modified E-glass fiber and iron(III) oxide particles with varying size. The particle sizes of 200 nm and <100 nm has been prepared by high energy ball milling and sol-gel methods respectively. To enhance better dispersion of particles and improve adhesion of fibers and fillers with epoxy matrix surface modification process has been done on both fiber and filler by an amino functional silane 3-Aminopropyltrimethoxysilane (APTMS). Crystalline and functional groups of siliconized iron(III) oxide particles were characterized by XRD and FTIR spectroscopy analysis. Fixed quantity of surface treated 15 vol% E-glass fiber was laid along with 0.5 and 1.0 vol% of iron(III) oxide particles into the matrix to fabricate hybrid composites. The composites were cured by an aliphatic hardener Triethylenetetramine (TETA). Effectiveness of surface modified particles and fibers addition into the resin matrix were revealed by mechanical testing like tensile testing, flexural testing, impact testing, inter laminar shear strength and hardness. Thermal behavior of composites was evaluated by TGA, DSC and thermal conductivity (Lee's disc). The scanning electron microscopy was employed to found shape and size of iron(III) oxide particles adhesion quality of fiber with epoxy matrix. Good dispersion of fillers in matrix was achieved with surface modifier APTMS. Tensile, flexural, impact and inter laminar shear strength of composites was improved by reinforcing surface modified fiber and filler. Thermal stability of epoxy resin was improved when surface modified fiber was reinforced along with hard hematite particles. Thermal conductivity of epoxy increased with increase of hematite content in epoxy matrix.

1. Introduction

Fiber reinforced polymer matrix composites have attractive applications in Structural, Automobile and Aerospace engineering. Adding fine fillers like Iron(III) oxide into high strength thermoset polymer like epoxy along with glass fiber improve mechanical strength and thermal stability [1,2]. Fiber reinforcing is a common way of strengthening polymers and addition of fillers also will serve substantial contribution in strength improvements [3,4]. Adding more volume of fiber may improve tensile and impact strength at the same time layer delaminating occurs under tensile, compression and shear loading. Composite strength can be improved by adding siliconized iron(III)oxide fillers along with fiber may increase mechanical strength without fiber delamination flaw [5,6]. To enhance maximum utilization of these composites controlled size of particles are very much important. The different particle sizes of 200 nm and <100 nm could be achieved by ball milling process and sol-gel method. High energy planetary ball milling was generally used since it is a cheaper way of producing low dimensioned particles [7]. Sol-gel method could also be used to produce nano scale particles in the range of 20 nm to 100 nm with controlled temperature, stirring time and mixing ratio. More uniform dispersion of particles in the resin matrix and high adhesion between fiber and matrix is required to yield good mechanical strength and thermal stability. To acquire homogeneous mixture of resin – fillers and improving adhesion between fiber and matrix many routes are available, in this chemical route is simpler and more feasible [8]. To achieve good dispersion of iron(III) oxide particles and adhesion of fiber into the epoxy resin matrix surface modification with an amino functional coupling agent called 3-Aminopropyltrimethoxysilane (APTMS) could be used. Cluster formation has reduced significantly because of surface modification and reaction between matrixes to iron(III) oxide can be improved by NH_2 functional group which is in silane coupling agent. The FTIR spectra result confirms the attachment of bi functional cou-

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pling agent in the treated iron(III) oxide particles with APTMS. Surface treated fibers are more adhere with resin matrix than the as received fibers because of chemical attachment between them. The conductive filler in the polymers serves the charge mobility in the presence of high frequency electric field hence more heat dissipation is possible because of the insulating property of epoxy matrix.

2. Experimental procedures

2.1. Materials

The epoxy resin used in the present study was a liquid diglycidyl ether of Bisphenol-A type (Huntsman India Ltd. Mumbai, Araldite LY556) with an equivalent weight per epoxide group of 195 g/mol having viscosity of 12,000 cps and density of 1.2 g/cm^3 at 25 °C. Triethylene tetramine (TETA, Huntsman India Ltd., Mumbai., HY951), a low viscosity aliphatic amine having viscosity of 20 cps and density of 0.98 g/cm^3 was used as a curing agent. 3-Aminopropyltrimethoxysilane (APTMS) was purchased from Sigma Aldrich. E-glass fiber continuous woven mat (0–90°) with density of 2.54 g/cm^3 was used as reinforcement. Ferric Chloride of molecular weight 169.8 g/mol and NaOH of molecular weight 40 g/mol were taken for sol-gel process and they were purchased from Merck india. ltd. Ball milled Iron(III) oxide particles with an average particle size of 800 nm, 200 nm and sol-gel prepared average 100 nm with density of 5.2 g/cm^3 were used as a filler to fabricate the hybrid polymer composite. All the chemicals and materials were used in as received condition without any post process treatments.

2.2. Sample preparation

2.2.1. Ball milling process

The as-received iron(III) oxide particles of diameter 800 nm were ball milled for 16Hrs. High energy planetary ball mill was used to reduce particle size by strain hardening principle. The powder to ball ratio was maintained as 1:15 and ball material used was tungsten carbide. The planetary mill speed was setup with 300 rpm throughout the milling time. Particle size and morphology of particles were monitored by particle size analyzer and scanning electron microscope for intervals (1 h, 2 h, 4 h, 8 h, 16 h) respectively. It was noted that a gradual decrement in particle size at the first 8 h and less reduction in last 8 h (8–16th h), this is because of in the initial stages particle size was high and more strain hardening occurred due to more collision of balls into the particles. This high strain hardening leads formation of small particles with fewer dimensions. Less dimensioned particles had less probability to getting hit by high energy balls, thus during last 8 h of milling no significant reduction has observed. Fig. 1 shows variation in particle size with different ball milled time of iron(III) oxide particles.

The scanning electron microscopy images of ball milled iron(III) oxide particles in Fig. 2 reveals that as milling time increases the size and shape of particles changed. It was observed that in Fig. 2(a) the particles were in needle structure and irregular, where as in Figs. 2(e and f) particles of reduced size and uniform spherical shape has been observed because of high milling time up to 8 h and 16 h respectively

2.2.2. Sol-gel process

Sol-gel is one of the easiest and low temperature synthesis methods in chemical route of preparing nano scaled materials [9,10]. Ferric Chloride ($FeCl_3$) of 1Mol and NaOH of 2 mol has been taken for sol-gel process. The sol of two metal atoms was prepared after constant stirring up to 1 h with 60 °C raising the temperature of sol up to 80 °C and allowed to evaporate the sol until dark brown gel was formed. The gel was kept in a silica crucible at 300 °C in

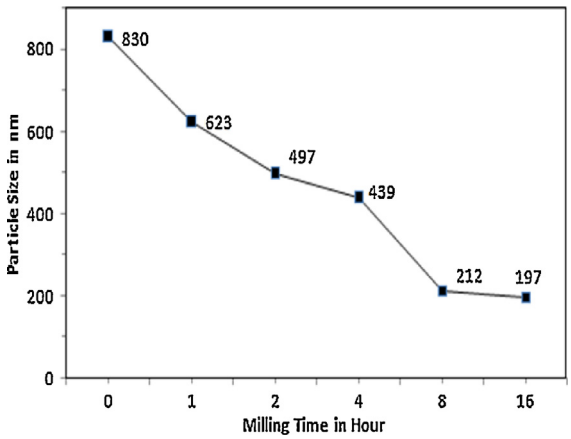

Fig. 1. Effect of milling time on particle size.

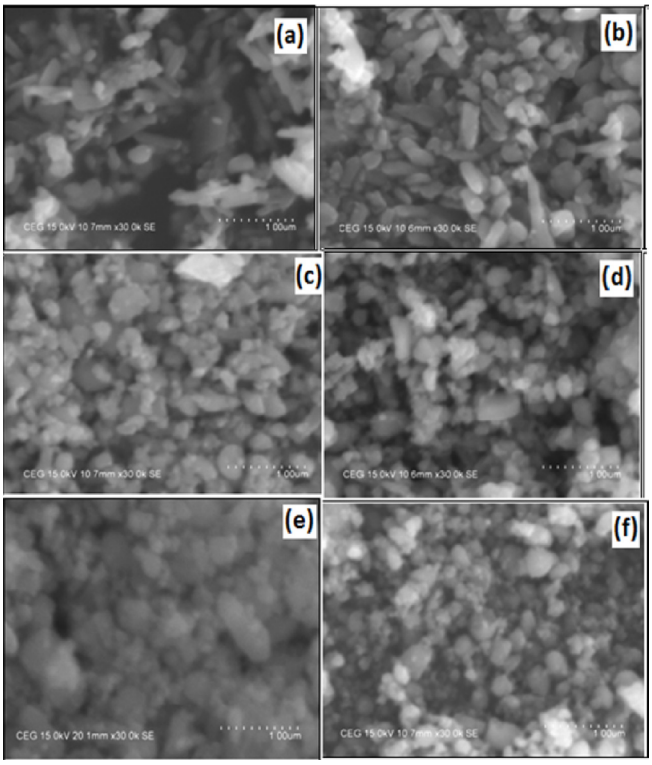

Fig. 2. SEM images of (a) 0 h, (b) 1 h, (c) 2 h, (d) 4 h, (e) 8 h, (f) 16 h ball milled iron(III) oxide particles.

hot oven for 4 h and then furnace cooled to room temperature thus nano scaled iron(III) oxide was formed. Eqs. (1) and (2) gives the nano iron(III) oxide formation.

$$FeCl_3 + 3Na(OH)_3 \rightarrow Fe(OH)_3 + 3NaCl \quad (1)$$

$$2Fe(OH)_3 \xrightarrow[-3H_2O]{\Delta} Fe_2O_3 \quad (2)$$

Crystal structure and form of iron(III) oxide was confirmed with X-ray diffraction peaks. The Fig. 4a and b shows XRD graph of ball milled and sol gel prepared iron(III) oxide particles. The strong peak at 33.2° (2θ) reveals the presence of rhombohedra α-fe_2o_3 with [h,k,l] pattern of (104) and other weak peaks of α-fe_2o_3 was appeared in 35.1° (110), 49.3° (024), 54.3° (116). Crystallite size of particles was found out using Debye-Scherer formula and the value was 42 nm for both ball milled and sol gel prepared iron(III) oxide. The Fig. 3 shows transmission electron microscopy image