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Original Article

Silane coupling agent for enhanced epoxy-iron oxide nanocomposite

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ABSTRACT

In this study, silane-treated Fe_2O_3 nanoparticles were successfully prepared using (3-aminopropyl) triethoxysilane (APTES). The chemical structure and morphology of the obtained nanoparticles were investigated by several analysis techniques including FTIR, XRD, TEM and DLS. Both of untreated Fe_2O_3 (IO) and silane-treated Fe_2O_3 (SIO) nanoparticles were used in the preparation of epoxy nanocomposites with 5% by weight fraction. FTIR and XRD approved that SIO was successfully prepared with highly crystalline structure. TEM and DLS indicated the good dispersion of treated nanoparticles in the nanocomposite matrix, also the average particle size of nanofiller decreased to ~200 nm after silane treatment. The dynamic properties for the prepared nanocomposites were studied using DMA and confirmed by nanoindentation technique. The results indicated that silane-treated nanoparticles can improve the hardness and *Tg* by 87.5% and 5 °C respectively at 5% weight fraction.

1. Introduction

Epoxy is considered as one of the greatest desired thermoset polymers. This results from its high mechanical and physical characteristics and highly cross-linked nature. Extensively, epoxy is being applied in several areas such as "constructions, car accessories, electrical insulators, marine and aerospace applications" [1]. Whilst the neat crosslinked epoxy has a brittle nature, low fatigue resistance and low toughness manner, this can restrain the growth of its applicable domain. Several attempts were actually suggested to enhance the physical, mechanical and thermal characteristics of epoxy through filling epoxy matrix with different nanofilling materials. Concerning this, nanofillers with many types and different grades such as "SiO_2, CNT, Al_2O_3, natural and synthetic nanofibrils, MMT and TiO_2" were loaded within epoxy matrix with different ratios to obtain improved nanocomposites [2]. For example, Phong et al. successfully fabricated micro and nano bamboo fibers using mercerization treatment followed by "mechanical extraction" technique that called "micro-grinding". When epoxy matrix was filled by 0.8 wt.% treated nanofibers, the obtained nanocomposite achieved enhanced "fracture toughness" by 84.6% [3].

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In fact, the two factors of "high viscous matrix" and "large specific surface area of nanofiller" can lead to bad dispersion of nanofiller within matrix. In this case, the collected and agglomerated nanofillers cannot enhance the different characteristics of the resulted nanocomposite. As well, these agglomerated ones may help and increase the start of cracking process that leads to premature deformation. Some attempts such as chemical and physical treatments have been studied to overcome this disadvantage. Adding of suitable coupling agents, using of surface coating and addition of some surfactants are some of these common treatments [4].

Actually, the surface treatment using a suitable coupling agent helps and improves the combination between the matrix and its filling material, the reinforcement process in this case will be highly effective [5]. The silane coupling agent, for example, such as methyltrimethoxysilane and aminopropyltriethoxysilane, has inorganic and organic ends. Silane reacts with the inorganic filler through its easily hydrolysable group meanwhile it attaches with the matrix through its organic end. So, silane can attach both organic matrix and inorganic filler together resulting in new filler–matrix interface [6,7]. The influence of chemical treatment for "aluminum powder" with "3-aminopropyltriethoxysilane" coupling agent on the epoxy characteristics was reported by Kim et al. [8]. In this study, the "tensile strength and modulus" for chemically treated composite were respectively enhanced by 12 and 9%, compared with neat epoxy.

Concerning current study, epoxy-iron oxide nanocomposites were formulated through filling epoxy matrix with iron oxide and silane-modified iron oxide nanoparticles. For studying the effect of treatment of filler with silane on the overall composite; microstructural, physical and mechanical characterizations were performed using FTIR, XRD, TEM, DLS, nanoindentation and DMA techniques.

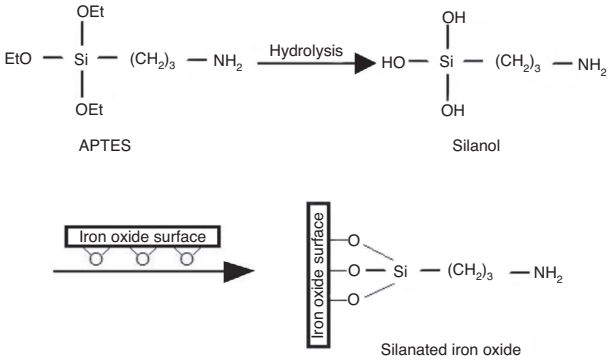
2. Materials and methods

2.1. Materials

Epoxy matrix (635-Thin epoxy resin-US Composites) with viscosity of 600 cps was used with its amine hardener as 2:1. Iron oxide (IO) nanoparticles, Fe_2O_3, was used as filler and aminopropyltriethoxysilane (APTES) was used as coupling agent; both were purchased from Sigma Aldrich.

2.2. Treatment of Fe_2O_3 nanoparticles

Firstly, 0.5 g of IO nanoparticles was dispersed into 400 ml of ethanol using ultrasonication for 10 min. The chemical treatment of iron oxide with silane was started by adding 2 ml of APTES dropwise to the dispersed solution of IO. Powerful stirring has been applied for this system and maintained for 24 h. The obtained treated material was then centrifuged, washed with ethanol-distilled water solution (1:1) three times and finally dried at 80 °C to obtain SIO as illustrated in Scheme 1.



Scheme 1 – Formation of silanated iron oxide nanoparticles.

2.3. Preparation of Fe_2O_3-epoxy nanocomposite

Epoxy matrix was filled with loading percentage of 5% using both IO and SIO nanofillers. Separately, IO and SIO were firstly mixed well into epoxy base and then were dispersed using ultrasonication for 10 min in ice bath. After addition of the hardener, the epoxy-IO and epoxy-SIO nanocomposites were kept for curing at room temperature for 7 days in teflon molds to avoid stacking.

2.4. Characterizations

2.4.1. Fourier transform-infrared

The spectral data of Fourier transform-infrared (FTIR) were detected within a wave number range of 400–4000 cm^{-1} using Nicolet IS-10 FTIR spectrophotometer-Thermo Fisher Scientific. The specimens were finely grinded followed by mixing along with KBr in the form of pellets.

2.4.2. X-ray diffraction

The crystallinity of treated and untreated samples was characterized using X-ray diffraction (wide angle XRD) analysis. The diffraction patterns were measured by Panalytical-X'pert-PRO with the Cu K-alpha source. The analysis was run though "1.54 Å wavelength, 40 kV voltage and 40 mA current". The 2θ range was 4–80° with a rate of 1°/min.

2.4.3. Transmission electron microscopy

The morphological features were examined by transmission electron microscopy (TEM) using high resolution JEOL-2100F TEM at 200 kV.

Measurements were conducted by preparing the sample in a diluted ethanol colloidal mixture which was sonicated for 15 min. A droplet of the dispersed solution has been positioned on the TEM grid. After drying, the grid was connected to the microscope and images were captured.

2.4.4. Dynamic light scattering

The "particle size distribution" for IO and SIO was statistically measured using Malvern dynamic light scattering (DLS). The specimen was firstly well dispersed and sonicated in deionized-water 10 min. The obtained dispersed solution was then subjected to DLS at 23 °C with a scattering angle of 90°.

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