Fabrication and Characterization of Epoxy Nanocomposites with Graphenes

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Summary. *Present research analyzed the manufacturing, using high speed rotational stirring and calendering as dispersion methods, of epoxy resins nanodoped with variable contents graphene (from 0.5 to 3.0 wt. %) of different kinds of graphenes characterized by their different particle size and number of graphenic layers. Produced nanocomposites were structurally, thermal and thermomechanically characterized. Effects of processing variables and graphene structural characteristics on nanocomposite properties have been evaluated.*

1 INTRODUCTION

During last 3-5 years, the interest of the international research community on the outstanding properties of graphene has grown displacing the research on other nanomaterials, such as carbon nanotubes. It is because graphene has superior electric and thermal conductivity, stiffness and strength, high specific surface area of up to 1800 m^2/g, jointly a high aspect ratio [1]. These properties open the possibility to apply them as a nanoreinforcement to improve physical and mechanical properties of polymers, being the epoxy resins among them.

However, as in the case of reinforcing epoxy resins with carbon nanotubes or nanofibres, it is necessary to reach a suitable dispersion of the nanofillers and to obtain a good interaction between the polymeric matrix and the graphene fillers. To meet these requirements, methods of dispersion must be developed, trying to avoid the reagglomeration phenomena which usually occur when nanofillers with high specific surface areas are dispersed into a polymer.

In this study, commercial graphenes with different grade of exfoliation and particle dimensions were dispersed into a bicomponent epoxy resin, using different level of reinforcement. Two dispersion methods were evaluated: high speed rotational stirring by means a toroidal stirrer and calendeing using a three rolls mill. The curing behavior and thermophysical properties of the graphene epoxy nanocomposites were characterized.

2 EXPERIMENTAL PROCEDURE

Nanocomposites were manufactured using an epoxy resin in which graphene nanoparticles were added. The epoxy resin used as matrix was a low viscosity resin called *Araldite LY556* that is cured with the addition of a hardener based on an aromatic amine with commercial name *Araldite XB3473*. Both components were provided by *Huntsman*. Four types of graphenes were studies, all of them supplied by *Graphene Supermarket*. Figure 1 resumes the main structural characteristics of theses nanoreinforcements. The manufacturing of nanocompostes with contents of 0.5, 1 and 3 wt. % of graphene nanoparticles were carried out in three steps: (1) dispersion of the nanoreinforcement into the epoxy resin; (2) addition of the

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hardener and degasification of the mixture; and (3) curing stage. Three dispersion methods were analyzed: i) high speed toroidal stirring with *Dispermat*®, ii) calendering with a three roll machine *EXAKT 80E* and iii) a combined method of previous ones.

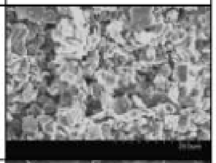
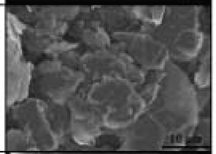
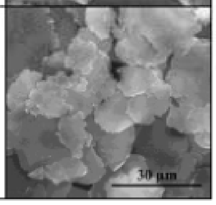
Type	Purity (%)	Specific Surface Area (m/g^2)	Average Flake Thickness (nm)	Average Particle Lateral Dimension (nm)	Materials	Image
A02	99.9	100	8 (20 – 30 layers)	550 (150 – 3000)	Dispermat 0.5 wt.%	
A03	99.2	80	12 (30-50 layers)	4500 (1500 – 10000)	Dispermat 0.5, 1, 3 wt.%	
A04	98.5	<15	60	3000 - 7000	Dispermat Calandra Disp + Calandra 0,5 wt.%	
C1	97	60	5 - 30	5000 – 25000	Dispermat Calandra Disp + Calandra 0,5 wt.%	

Figure 1: Denominations and structural characteristics of graphene nanoparticles used for nanocomposites manufacturing. Dispersion methods are also indicated.

The nanocomposites were observed using a *Nova Nano SEM230* field emission scanning electron microscope (FEG-SEM) and a *Philips Tecnai 20T* transmission electron microscope (TEM). The nanocomposites thermomechanical properties were evaluated using dynamic mechanical thermal analysis (DMTA). A *DMA Q800 V7.1* machine from TA Instruments operating in single cantilever mode with an oscillation frequency of 1.0 Hz. was used for this purpose, determining both the storage modulus (E_0) and the glass transition temperature (T_g). Thermogravimetric analysis (TGA) measurements of the cured nanocomposites were also carried out in *Setaram mod Setsys 16/18* thermobalance.

3 RESULTS

Highest values of E_0 were obtained when graphene nanoparticles which had the highest specific surface and exfoliation grade (C1) were added. Beside, the combination of both dispersion methods (Dispermat plus calandering) provided the best thermomechanical properties.

REFERENCES

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