Characterization of epoxy/graphene nanocomposites by dynamic mechanic analysis using Time-Temperature superposition.

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Introduction

Epoxy Systems enriched with carbon nanomaterials have been proposed for applications such as high performance materials. These resins are described by the presence of epoxy rings capable of forming a network through curing reactions with a variety of agents. The modification of carbon nanomaterial has been proposed with the aim of improving the interface filler-polymer without harming the reticulation reaction between the resin and the curing agent, producing materials with different properties and applications. In this work the system composed of resin epoxy bisphenol A diclycidyl ether and the curing agent triethylenetetramine (DGEBA-TETA) was used for the nanocomposites preparation with graphene oxidized (GO), reduced graphene (RGO) and functionalized graphene with 4,4'–Methylenebis (phenyl isocyanate), MDI. These materials were characterized by Raman spectroscopy, X-ray photoeletronic spectroscopy (XPS), and atomic force microscopy (AFM). The mechanical properties of the nanocomposites were investigated by means of master curves of relaxation obtained by dynamic-mechanical analysis (DMA) and the Arrhenius' model of time-temperature superposition (TTS)[1].

Results e Discussion

The table 1 summarize the results of characterization of graphene and functionalized graphene. The C/O ration from XPS, the D/G ratio from Raman spectroscopy, and the thickness obtained by profile curves in AFM microscopy shows that graphene has been successful obtained and presents a few layers[2].

Table 1. Characterization of graphenes.

Graphene	C/O	D/G	Thickness
GO	0.43	1.78	0.97 nm
RGO	2.06	1.46	0.70 nm
GO-MDI	--	1.72	2.50 nm
RGO-MDI	--	1.48	2.30 nm

The graphene-based nanocomposites were produced with 0.5% wt. of graphene, and DGEBA/TETA with 14 phr. The mechanical characterizations by DMA shows an improvement in the glass transition (Tg) greater than 10°C.

The sweep of frequency was executed and, with the Arrhenius' model TTS, the master curves were plotted in the reference temperature of 40°C, figure 1 (a). The master curve shows that nanocomposites presented mechanical stability better than neat epoxy. The master curves can also express the activation energy (Ea) of glass transition. The results shows an improvement about 55% in the Ea in the nanocomposites. The behavior of Tg with frequency were obtained with the master curves data, figure 1 (b). That results shows that Tg is dependent of frequency besides (and) they indicate that graphene-MDI composites has a most important increases on Tg.

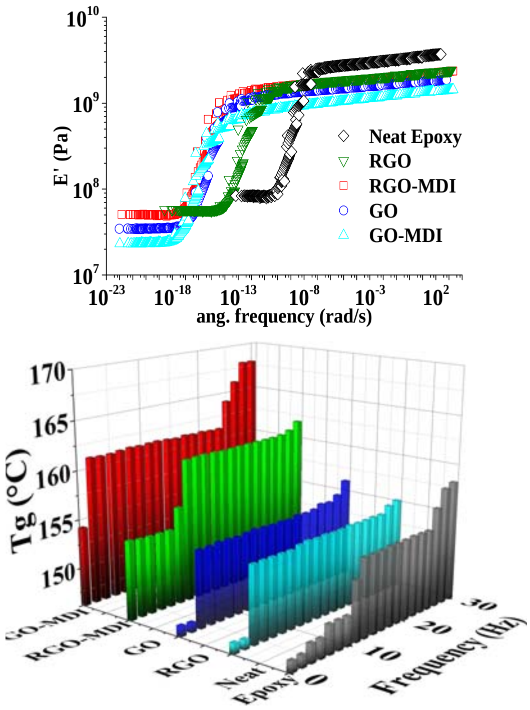

Figure 1. Master curves of nanocomposites (a), and the curves of Tg as a function of frequency (b).

Conclusions

The graphenes and functionalized graphenes were successful produced. The dynamical mechanical analysis associated to time-temperature models is adequate to promote the complete mechanical characterization of Epoxy/graphene nanocomposites.

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[1]Li R. Time-temperature superposition method for glass transition temperature of plastic materials 2006;278:36–45.
[2]Stankovich S and Ruoff RS. Synthesis and exfoliation of isocyanate-treated graphene oxide nanoplatelets. Carbon N Y 2006;44:3342–7.