ADVANCED TRIFUNCTIONAL EPOXY – SWNTs COMPOSITE MATERIALS. PREPARATION AND CHARACTERIZATION

J.M. González-Domínguez[1], P. Castell[1], A. Ansón[1], A. Benito[1], W. Maser[1], M. Naffakh[2], A. M. Díez-Pascual[2], M. A. Gómez[2], M.T. Martinez[1]*.
1) Instituto de Carboquímica (CSIC), C/ Miguel Luesma Castán nº4 50018, Zaragoza, Spain
2) Instituto de Ciencia y Tecnología de Polímeros (CSIC), C/ Juan de la Cierva 3, 28006 Madrid, Spain
* Corresponding author's e-mail : mtmartinez@icb.csic.es

INTRODUCTION

Epoxy resins represent one of the most widely used kind of polymer, being a key element in material science and engineering, specially in the field of high performance materials, coatings and composites **[1]**. The wide variety of epoxy systems available, with very different crosslinking degrees and thus very different physical performances, makes these thermosetting polymers extremely suitable for purposes such as structural, medical or aerospatial. From a few years on, a growing interest has emerged in multifunctional epoxies, specially in trifunctional resins **[2]**. Although epoxy resins are inherently brittle, the main advantage these materials present is a quite high modulus, stiffness and thermal stability, and also for being capable of toughening throughout the addition of other components. Thermoplastic polymers, nanoclays, fibres and carbon nanotubes represent the most frequently used reinforcers.

Carbon nanotubes lay in the forefront of nanoscience researching because of their unique properties and outstanding performance. They are known to be one of the best reinforcers in composites field **[3]**, despite the drawbacks related to the proper integration, dispersion and adhesion along the polymer matrix. Single Walled Carbon Nanotubes (SWNTs) are preferred in composites where mechanical performance is wanted to improve substantially. Both covalent and non-covalent functionalization of SWNTs have been experimented as key techniques for disentangle their bundles and improving the interfacial adhesion to the matrix. In the non-covalent functionalization, block copolymers have been shown as excellent promoters of wetting and adhesion, offering a suitable alternative in carbon nanotubes interfacial union engineering. The integration of polymer wrapped carbon nanotubes into polymeric matrices may lead to a more homogeneous distribution of the reinforcer. Other features of the as-prepared nanocomposites, specially involving physical performance, represent critical issues.

EXPERIMENTAL AND RESULTS

We have previously reported **[4]** the use of a block copolymer (Pluronic F68) to manufacture highly unbundled (Fig. 1) dispersions of wrapped arc discharge SWNTs. Nanocomposite materials based on Triglycydyl-p-aminofenol (TGAP) trifunctional epoxy and 4,4'-diaminodifenilsulfone (DDS) curing agent were prepared with different contents of Pluronic wrapped SWNTs (0.1, 0.25, 0.50, 1.0 and 2.0 wt% respectively), which were successfully integrated into an epoxy matrix, causing also improvements in the curing kinetics.

Wrapped SWNTs were integrated into the epoxy matrix by direct mixing on the epoxy precursor, optimizing the experimental process of heating and sonication, with further optimized incorporation of the curing agent. Mixing time and temperature of TGAP + DDS system were optimized using differential scanning calorimetry (DSC). Isoconversional analysis **[5]** on the different SWNTs-Pluronic reinforced epoxy samples was carried out throughout dynamic heating scans at different rates (2, 5, 10 and 20 ºC/min). Results indicated that Pluronic remarkably affects kinetics in the extreme stages of the curing reaction by easing mobility of species, allowing the addition of SWNTs without worsening processability and also promoting a much more homogeneous distribution. This was also confirmed by scanning electron microscopy (SEM). Thermogravimetric analysis (TGA), Thermally Programmed Desorption (TPD) and Mass Spectrometry (MS) were also employed to check stability and degradation profile of the neat resin and nanocomposites.

The effect over the mechanical properties in the resulting nanocomposites was checked by dynamical mechanical analysis (DMA), concluding that there is no negative effect on the storage modulus with the presence of Pluronic in the nanocomposite, being even highly beneficial only at very low loadings. Neat increases in the SWNTs-Pluronic reinforced nanocomposites storage moduli of ~30% with respect to the bare resin were found. Electrical conductivity was substantially raised, from $\sim 10^{-13}$ (neat resin) to $\sim 10^{-7}$ S/cm, surpassing the percolation threshold at 0.1 wt% of wrapped SWNTs.

References:

[1] C.A. May, Epoxy resins. Chemistry and technology, New York: Marcel Dekker (1988).
[2] F. Carrasco, P. Pagès, T. Lacorte, K. Briceño, J. Appl. Polym. Sci. **98**, 1524 - 1535 (2005).
[3] J. N. Coleman, U. Kanh, W. J. Blau, Y. K. Gun'ko, Carbon, **44** (2006) 1624 – 1652.
[4] J.M. Gonzalez-Dominguez, P. Castell, A. Anson, W.K. Maser, A. Benito, M.T. Martinez, accepted in the *Journal of Nanoscience and Nanotechnology*.
[5] S. Vyazovkin, J. Comput. Chem. 18, 393-402 (1997)

Figures:

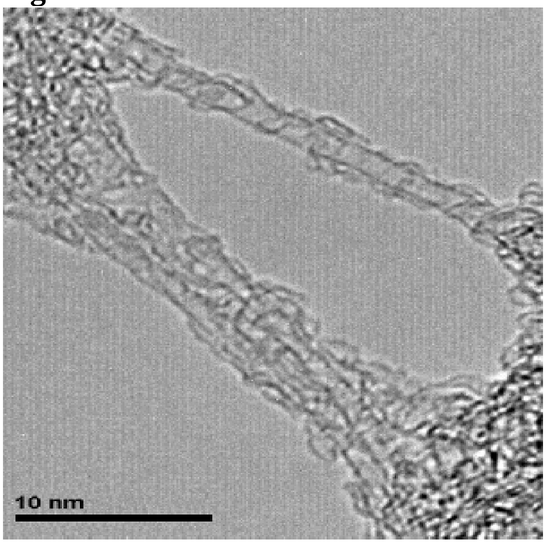

Figure 1: Transmission electron microscopy image of Pluronic wrapped arc SWNTs