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Carbon nanotubes-based nanohybrids for multifunctional nanocomposites

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ABSTRACT

In the present work, nano-hybrids based on carbon nanotubes (CNTs) bearing immobilized, either through covalent linkage and physical absorption, commercial anti-oxidant molecules have been formulated and used as nanofillers in Ultra High Molecular Weight Polyethylene (UHMWPE), aiming at preparing multifunctional nanocomposites. The effective immobilization of the anti-oxidant molecules has been probed by spectroscopic and thermogravimetric analyses. The study of the morphology and the rheological behaviour of the nanocomposites show that the immobilization of anti-oxidant molecules onto the CNTs surface is beneficial for the state of the polymer/nanoparticles interfacial region. Additionally, the study of the nanocomposites thermo-oxidative behaviour reveals that the nano-hybrids are able to exert a remarkable anti-oxidant action which is related to the strong interactions established between the anti-oxidant molecules and the CNTs, resulting in the formation of structural defects onto the CNTs surface and in the consequent amplification of the intrinsic CNTs radical scavenging activity.

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1. Introduction

Recently, significant efforts have been directed toward the formulation of multi-functional polymer-based nanocomposites containing hybrid nanoparticles (Arrigo et al., 2016a; Jie et al., 2015). In particular, carbon nanotubes (CNTs) have received a great attention as nanofillers for polymer-based complex systems, due to their unique combination of mechanical, electrical and magnetic properties (Coleman et al., 2006; Spitalsky et al., 2010). Furthermore, the CNTs surface can be easily modified with several functional groups carrying specific functionalities through different strategies, such as defect (Mawhinney et al., 2000) and covalent

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(Yang et al., 2007) functionalization. Interestingly, a promising strategy to modify the CNTs surface without compromise their structural integrity involves the non-covalent functionalization of CNTs with molecules showing a strong affinity with graphitic surfaces of CNTs (Nativ-Roth et al., 2007; Chen et al., 2008). An interesting paper by Pan et al. (Pan and Xing, 2008) reports the different mechanisms of adsorption of organic molecules on CNTs; in the case of aromatic moieties, the physical adsorption is believed to be due to the π - π electron coupling (Lin and Xing, 2008, Wang et al., 2010) between adsorbate molecules and carbon nanotube surfaces and this hypothesis is corroborated by experimental study and density functional theory calculations (Mallakpour and Soltanian, 2016). Moreover, recent studies (Yu et al., 2016) show that the π - π interactions between aromatic compounds and graphitic surfaces increased with increasing aromatic rings in aromatic molecules.

The modification of the CNTs surface, besides provide them with new functionalities, helps to enhance the state of the polymer/CNTs interfacial region, bringing about the obtainment of nanocomposites with superior properties, suitable for advanced applications (Arrigo et al., 2016b).

Recent researches have shown that CNTs are able to exert an excellent anti-oxidant action against the thermo- and

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photo-degradative processes of polymeric materials (Watts et al., 2003; Shi et al., 2011; Dintcheva et al., 2016a). This intriguing property of CNTs seems to be related mainly to a radical scavenging mechanism (Galano, 2008; Galano et al., 2010); specifically, CNTs, similarly to other carbon-based nanostructures such as graphene and fullerenes, are able to act as radical traps, reacting with the macro-radicals formed during polymer degradation (Martinez-Morlanes et al., 2012; Dintcheva et al., 2015a). The radical scavenging activity of CNTs is related to their acceptor-like electronic properties: due to the presence of lattice defects, CNTs possess acceptor-like localized states which are invoked as the main responsible for the radical termination (Dintcheva et al., 2015b). Furthermore, the UV stabilizing action of CNTs has been attributed to their photo-induced oxidation that occurs when they are exposed simultaneously to UV light and oxygen. Owing to this mechanism, the formation of locally electron-deficient and electron-rich regions onto the CNTs outer surface occurs. In this way, the oxygen molecules are adsorbed onto the formed electron-rich zones and are no longer available for polymer oxidation (Savage et al., 2003; Guadagno et al., 2010).

In recent studies reported in literature, the radical scavenging activity of CNTs has been amplified through the immobilization of molecules carrying anti-oxidant function onto the CNTs outer surface, considering two different approaches: covalent grafting and physical absorption (Arrigo et al., 2015; Dintcheva et al., 2014; Dintcheva et al. 2016b). In particular, natural stabilizing molecules, such as vitamin E and quercetin, have been physically immobilized onto the CNTs outer surface, and the obtained multi-functional fillers significantly improved the thermo-oxidation stability of a host polymeric matrix. Apart from the stabilizing activity of the functional moieties, this result was ascribed to an amplification of the inherent radical scavenging activity of CNTs, due to the strong interactions established between the carbon atoms of the CNTs surface and the immobilized molecules, which promote the formation of CNTs structural defects.

In this work, hybrid nanoparticles consisting of CNTs and a hindered phenol commercial anti-oxidant have been formulated through two different strategies: physical immobilization and covalent grafting. The nano-hybrids have been used as nanofillers in UHMWPE-based nanocomposites, with the dual aim to improve the state of polymer/nanoparticles interface and to enhance the nanocomposites thermo-oxidative stability.

2. Materials and methods

2.1. Materials

The UHMWPE is a commercial grade purchased by Sigma-Aldrich. It has weight-average molecular weight $3 \div 6$ MDa, softening point T = 136°C (Vicat, ASTM D 1525B), melting point T_m = 138 °C (determinate by DSC) and density 0.94 g/mL at 25 °C.

Multiwalled Carbon Nanotubes, CNTs, were prepared by the typical Chemical Vapor Deposition (CVD) protocol, using ethylene as carbon source (Dintcheva et al. 2016a). The purification was performed with 50% aqueous sulfuric acid, obtaining Carbon Nanotubes with outer diameters ranging between 14 and 20 nm, inner diameters in the range of 2–5 nm, length $1 \div 10 \mu$ m, and purity >98 wt%. After the purification process the content of carboxylic groups was estimated to be 0.5 $\div 1\%$ by means of XPS analysis and acid/base titration.

Irganox 1010 (Pentaerythritol Tetrakis(3-(3,5-di-tert-butyl-4-h ydroxyphenyl)propionate)), Irg1010, is a is an ex. Ciba[®] Specialty Chemicals (BASF).

All chemical reagents have been purchased by Sigma-Aldrich[®] and used as received.

2.2. CNTs functionalization

The Irg1010-*f*-CNTs have been obtained through a procedure summarized in Fig. 1. In particular, 350 g of HO-alkyl-*f*-CNTs, prepared following a procedure reported elsewhere (Dintcheva et al., 2016b), have been dispersed in a solution of Irg1010 (1.0 g) and Potassium *tert*-butoxide (0.21 g) in 150 ml of DMSO. The resulting mixture was sonicated in a ultrasound bath (240 W, 2.5 Lt) for 3 min, and then it was stirred at 80 °C for 24 h. After that, the suspension was filtered on a sintered glass filter and then the Irg1010-*f*-CNTs (**2**) were carefully washed with hot methanol, and finally dried at 90 °C overnight.

To prepare lrg1010@CNTs, lrg1010 molecules have been immobilized onto the outer surface of the CNTs as follow (see Fig. 1): 0.4 g of bare CNTs were dispersed in a solution of lrg1010 (0.2 g) in 30 ml of THF. The resulting suspension was sonicated in a 2 L ultrasonic bath (power 260 W) for 30 min to allow for the physical absorption of the anti-oxidant onto the CNTs. Then, the solute was filtered on a sintered glass filter and then the lrg1010@CNTs (**3**)



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