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Purification of Single Walled Carbon Nanotubes based on Fenton's Chemistry

S. D. F. Brandão[(1)*], A. P. C. Teixeira[(1)], D. M. Andrada[(1)], Regis F. Gontijo [(1)],
C. Fantini [(2)], A. P. Santos[(1)] and C. A. Furtado[(1)]

(1) Centro de Desenvolvimento da Tecnologia Nuclear - Belo Horizonte – Minas Gerais - Brazil
(2) Universidade Federal de Minas Gerais - Belo Horizonte – MG - Brazil
* sdfb@cdtn.br, clas@cdtn.br

Abstract – We have applied a "green" and scalable method based on Fenton's chemistry in order to purify several carbon nanotube samples, which were synthesized by arc discharge or chemical vapor deposition (CVD) using different transition metals (Fe, Co, Mo, Ni) as catalyst. Scanning electron microscopy (MEV), energy dispersive X-ray spectroscopy (EDS), Raman scattering, infrared spectroscopy (IR) and thermogravimetry (TG) have been used to determine the composition and purity degree of carbon nanotube samples before and after the purification process. In general, the results showed increase of the carbon content, but optimization is needed taking into account each catalyst metal and synthesis protocol.

Carbon nanotubes are a very promising material for applications in Nanotechnology. Several processes for making carbon nanotubes are being scaled up to meet the growing demand induced by such applications, but a unfortunate outcome of them is that the product is not pure. Residual growth catalyst, amorphous carbon and ceramic matrices are the most common impurities, being the real composition of the samples dependent on the synthesis route. Hence, post-synthesis purification protocols that enable to obtain high-purity material with minimal metal residues are strongly required. In this work, we have applied a "green" and scalable method based on Fenton's chemistry [1], highly selective toward the removal of the carbon-coated iron nanoparticles [2], in order to purify commercial (Carbolex®, HiPCO®, CoMoCAT®) and homemade (arc-CoNi and CVD-FE/MgO) single walled carbon nanotube (SWNT) samples, which were synthesized by chemical vapor deposition (CVD) or arc discharge processes using different transition metals (Fe, Co, Mo, Ni) as catalyst. Scanning electron microscopy (MEV), energy dispersive X-ray spectroscopy (EDS), Raman scattering, infrared spectroscopy (IR) and thermogravimetry (TG) have been used to determine the composition and purity degree of carbon nanotube samples before and after the purification process. In general, the relative percentage in weight of carbon nanotubes increased for all samples after purification. However, as expected, the formation of hydroxyl radicals from the acid solution of hydrogen peroxide seems not occur in presence of Ni and them the reduction of amorphous carbon and catalyst metal for the arc discharge SWNT samples was promoted by the direct reaction with H_2O_2 and HCl, respectively. The introduction of Co in the arc-CoNi sample completely changed the oxidation mechanism. Cobalt participates of oxi-reduction reactions and favors the oxidation of the impurities via formation of OH radicals. The reaction under strong conditions promoted the creation of defects and functionalization. For the SWNT samples synthesized by CVD, the oxidation also occurred via OH radicals. The creation of defects was clearly evidenced for HiPCO® samples. For CoMoCAT® and CVD-FE/MgO samples, the presence of a ceramic matrix supporting the catalyst blocked the removal of carbon and metal impurities. The results show that optimization is needed for all samples taking into account each catalyst metal and synthesis protocol.

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