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Functionalization of Multiwalled Carbon Nanotubes with alquilamines

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Abstract – In this work the functionalization of multiwalled carbon nanotubes (MWCNTs) with alquilamines (octadecylamine and dodecylamine) were carried out. Functionalization of MWCNTs was achieved by oxidation and acylation of the MWCNTs, followed by the peptidic bond formation between the carbonyl chloride and the amine groups. The covalent bond formation was characterized by Fourier transform infrared spectroscopy, and the morphology of the MWCNTs was observed by scanning electron microscopy. The solubilization was tested in different solvents.

Carbon nantoubes posses a number of unique properties and have been the subject of intense experimental and theoretical attention as materials that have great potential for use in emerging nanotechnologies. However, the application of carbon nanotubes in composites, preparation of films and microfibers, and the production of electronic, optical and optoelectronic components and devices often require nanotube functionalization [1].

The main approaches to carbon nanotube functionalization include carboxylation via acid treatment. So, the grafted carboxyl groups can be easily converted to carbonyl chloride groups via reaction with thionyl chloride ($SOCl_2$), which can then be further reacted with amine groups to form peptidic bonds. Most research effort has been concentrated on the functionalization and solubilization of single-wall carbon nanotubes. The processes with the participation of MWCNTs have been essentially unexplored.

In this work functionalization of MWCNTs with alquilamines were carried out. Functionalization of MWCNTs was achieved by using nitric and sulfuric acid mixture as oxidant. Acylation of the resulting carboxyl groups were performed by refluxing an excess of $SOCl_2$, followed by the peptidic bond formation between the acyl and the amine groups from octadecylamine (ODA) and dodecylamine (DDA). The covalent bond formation was characterized by Fourier transform infrared spectroscopy (FTIR), and the morphology of the MWCNTs was observed by scanning electron microscopy. The solubilization was also tested in different solvents by registering the electronic absorption spectrum.

The FTIR spectra of the MWCNTs and the carboxylated MWCNTs were seen to differ little in the position and intensity of IR bands. The main difference between then was the appearance of a band at 1710 cm^{-1}, which was assigned to the carbonyl from carboxylic acid in the oxidized carbon nanotubes. The position of the carbonyl band, was indeed used to characterized the formation of carbonyl chloride, which appeared as a doublet band in the range of 1730-1780 cm^{-1}, and the amide formation, which appeared at ~1650 cm^{-1} in the FTIR spectrum of the final product. The morphology of the resulting product was compared with the starting one, and no significant destruction of the MWCNTs was observed. The solubility of the MWCNTs was changed according to the treatment done. While the carboxylated MWCNTs were soluble in polar solvents like ethanol, the ODA- and DDA-functionalized MWCNTs were soluble in less polar solvents like chloroform, tetrahydrofuran and dichloromethane. The solubility values of the amidated MWCNTs agreed with the values reported in literature only approximately, and can be explained by the degree of functionalization, which on his turn, depends on the process conditions.

[1] N. T. Hung, I. V. Anoshkin, A. P. Dementjev, D. V. Katorov, E. G. Rakov, Inorganic Materials 44 (2008) 219-223.