CHARACTERIZATION AND ENHANCEMENT OF ELECTROCHEMICAL PROPERTIES OF MWCNT/POLYSULFONE COMPOSITE MODIFIED SCREEN-PRINTED ELECTRODES

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The Polysulfone(PSf)/Multi-walled Carbon Nanotubes (MWCNT) composites were prepared by the phase inversion method as previously reported [1,2]. Here we describe a profound characterization concerning the composite construction as well as the thermal, electronic, conductive, morphological, physical, chemical and electrochemical properties.

When polymer is mixed up with CNT suspension, a layer of ≈ 20 nm of polymer coats every single carbon nanotube separately as observed by SEM and TEM. The main difference (vs graphite composites) in the morphology structure relies on the interaction within the polymer matrix: whilst PSf wraps the MWCNTs separately in the PSf/CNT composite, the polymer glue the big graphite particles with a very low uniformity distribution.

Electronic properties of the composite were enhanced as observed by Atomic Force Microscopy. Thermo gravimetric analysis showed that the thermal stability of CNT composite was 8°C higher than graphite counterparts. Moreover, this nanocomposite was printed onto screen-printed electrodes, showing higher sensibility, lower resistance and electrocatalytic effect when compared with graphite composite modified electrodes. Impedance Spectroscopy was an interesting tool for the electrochemical resistance characterization of the composites deposited onto screen-printed electrodes. The electrochemical detection of some biomarkers as hydrogen peroxide, ascorbic acid and ferricianide was studied.

In this characterization work, we can conclude that the good interaction between the polymer and nanotubes gives some improved characteristics to the composite and therefore it can be also used as electrochemical platform due to its high electron transfer properties.

The electrochemical activation of multiwall carbon nanotubes (MWCNT) (at potentials of 1.5-2.0 V *vs.* Ag/AgCl for 60-360 s) results in significantly increased electrochemical and catalytic properties. This increase is due to introduction of dramatic wall defects exposing edge planes of MWCNT as well as the diminution of the PSf coating as it was observed by high resolution transmission electron microscopy. X-ray photoelectron spectroscopy (XPS), Raman spectroscopy and electrochemical impedance spectroscopy were used to gain deeper understanding the phenomena. XPS revealed that electrochemical activation of carbon nanotubes leads to relative increase of presence of carboxyl groups while relative presence of carbonyl and alcohol/ether groups decreases.

Electrochemical activation of carbon nanotubes was studied in previously [3, 4], but they presented only electrochemical data and it did not bring any spectroscopic insight why such enhancement happens until Pumera investigations [5]. Moreover, we did not find previous work based on the electrochemical activation of carbon nanotube-based composites. This electrochemical activation was applied to a screen-printed electrode used for cyclic voltammetry of important biomakers.

The aim of this article is to demonstrate that electrochemical activation of carbon nanotubes results into introduction of defects to carbon nanotube walls which lead to higher electroactive sites density and consequently to faster observed heterogeneous electron transfer rate k^0_{obs} . We characterize electrochemically activated carbon nanotubes by cyclic voltammetry, electrochemical impedance spectroscopy, Raman spectroscopy, high resolution transmission

electron microscopy, Scanning electron microscopy and X-ray photoelectron spectroscopy to have insight to the mechanism of such activation.

References:

- [1] Sanchez, S., Pumera, M., Fabregas, E. Biosens. Bioelectron. 23 (2007) 332.
- [2] Sanchez, S., Pumera, M., Cabruja, E., Fabregas, E. Analyst 132 (2007) 142.
- [3] Musameh, M., Lawrence, N. S., Wang, J.. Electrochem. Commun. 7 (2005) 14.
- [4] Wang, J., Li, M., Shi, Z., Li, N., Gu, Z. Anal. Chem. 74 (2002) 1993.
- [5] Pumera, M. Langmuir. (2008). On revision.

Figures:

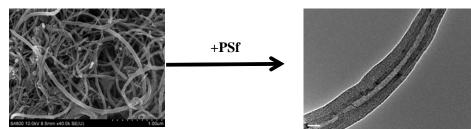


Fig.1. (left) SEM image of CNT powder and (right) TEM of a single CNT with Polysulfone

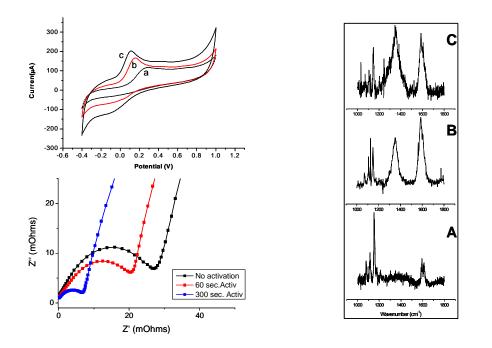


Fig.2. Cyclic voltammograms of ascorbic acid for MWCNT/PSf-modified sensor and Impedance Spectra for ferricianide, a) no activation, b) activation at +1.75 V for 60 sec and c) for 300 sec. Fig.3. Raman spectroscopy of: A) PSf, B) MWCNT/PSf composite and C) MWCNT/PSf composite after activation of 60 s at +1.75 V.