



Extended Abstract

## Chemical Synthesis of Multi-Walled Carbon Nanotubes and Their Functionalization with Carboxylated Groups <sup>†</sup>

Madalina Elena David 1,2,\*, Rodica-Mariana Ion 1,2, Ramona Marina Grigorescu 1, Lorena Iancu 1,2, Elena Ramona Andrei 1, Raluca Somoghi 1, Adriana Nicoleta Frone 1 and Raluca Maria Stirbescu 3

- National Institute for Research & Development in Chemistry & Petrochemistry–ICECHIM, 202 Splaiul Independentei, 060021 Bucharest, Romania; rodica.ion@icechim.ro (R.-M.I.); ramona.grigorescu@icechim.ro (R.M.G.); lorena.iancu@icechim.ro (L.I.); ramona.andrei@icechim.ro (E.R.A.); raluca.somoghi@icechim.ro (R.S.); adriana.frone@icechim.ro (A.N.F.)
- Valahia University, Doctoral School of Materials Engineering Department, 13 Aleea Sinaia, 130004 Targoviste, Romania
- <sup>3</sup> Valahia University of Targoviste, Institute of Multidisciplinary Research for Science and Technology, 13 Aleea Sinaia, 130004 Targoviste, Romania; stirbescu.raluca@icstm.ro
- \* Correspondence: madalina.david@icechim.ro
- † Presented at the 16th International Symposium "Priorities of Chemistry for a Sustainable Development" PRIOCHEM, Bucharest, Romania, 28–30 October 2020.

Published: 11 November 2020

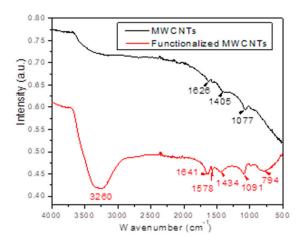
Keywords: carbon nanotubes; nanomaterials; chemical synthesis

Carbon nanotubes (CNTs) have attracted attention, due to their structures as well as their ability to present multiple walls. CNTs can be synthetized by several methods, such as arc-discharge, chemical vapor deposition, laser ablation and electrolysis. All these methods lead to CNTs with different diameters, lengths and numbers of layers, but these methods require high costs of production, high temperatures and high pressures, and most of them require repeated procedures for their purification after obtaining. As an alternative to a avoid these disadvantages, chemical techniques seem to be a good option to obtain CNTs easily (at low temperatures and without applying pressure) and inexpensively [1].

Multi-Walled Carbon Nanotubes (MWCNTs) were prepared by chemical synthesis using graphite microparticles, concentrated sulfuric acid, fuming nitric acid and potassium chlorate. The synthesis parameters were fixed based on previous work [2]. In order to improve the solubility of the MWCNTs in organic solutions, acid treatment was used (H<sub>2</sub>SO<sub>4</sub> (95%) and HNO<sub>3</sub> (65%) in a ratio of 3:1) by functionalizing the surfaces of CNTs with negatively charged functional groups (MWCNTs-COOH). The obtained materials were investigated by Fourier-transformed infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM) and atomic force microscopy (AFM).

The research focused on analyzing both the MWCNTs obtained by this new method and the functionalized MWCNTs. The major functional groups of the MWCNTs and functionalized MWCNTs were identified by FTIR analysis (Figure 1). Moreover, in order to estimate the functionalization, the same quantity of MWCNTs was dispersed in water, before and after functionalization, for 2 h in an ultrasonic bath and afterwords visually observed to evaluate their suspension stability (Figure 2).

Proceedings 2020, 57, 45 2 of 2



**Figure 1.** FTIR spectra of the Multi-Walled Carbon Nanotubes (MWCNTs) and functionalized MWCNTs.



Figure 2. Aqueous dispersion of (1) pure MWCNTs and (2) functionalized MWCNTs.

By using this chemical route, pure MWCNTs were synthetized (sustained by FTIR and EDAX), with a diameter between 9 nm and 43 nm and 500–600 nm in length, as proven by TEM and AFM analysis. The functionalization of these MWCNTs was also successfully done, which was confirmed by FTIR and aqueous dispersion of the nanotubes.

**Acknowledgments:** This paper was supported by a grant of the Romanian Ministery of Research and Innovation, CCCDI—UEFISCDI, contract number 51 PCCDI/2018, within PNCDI III and by project no. PN.19.23.03.01, contract no. 23N/2019 within the Nucleu Program.

## References

- David, M.E.; Ion, R.-M.; Grigorescu, R.M.; Iancu, L.; Andrei, E.R. Nanomaterials Used in Conservation and Restoration of Cultural Heritage: An Up-to-Date Overview. *Materials* 2020, 13, 2064–2087, doi:10.3390/ma13092064.
- 2. Ali, B.; Biak, D.R.A.; Sapuan, S.M.; Zaidan, A.W.; Yusoff, H.M.; Masrinda, T.S. Preparation of Carbon Nanotubes via Chemical Technique (Modified Staudenmaier Method). *Nanosci. Nanotechnol. Asia* **2017**, 7, 113–122, doi: 10.2174/2210681206666160711161421.

**Publisher's Note:** MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



© 2020 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).