

Carbon Nanostructures Synthesized by Chemical Reaction Using Rongalite and Polyethyleneimine as Complex Agents

J.A. González¹, R. C. Carrillo-Torres¹, M. E. Alvarez-Ramos¹ and S. J. Castillo²

¹ Departamento de Física, Universidad de Sonora, Hermosillo, Sonora, México.

² Departamento de Investigación en Física, Universidad de Sonora, Hermosillo, Sonora, México.

Carbon atoms form various allotropes with different physical and chemical natures originated from the variety of s-p orbital hybridization [1,2]. Carbon is a remarkable element showing a variety of stable forms ranging from 3D semiconducting diamond to 2D semi-metallic graphite to 1D conducting and semiconducting carbon nanotubes to 0D fullerenes, in this work we present a synthesis route to carbon with hierarchical morphology on the nanoscale.

Carbon nanostructures have been synthesized by chemical reaction in aqueous solution using saccharose, deionized water, rongalite and sulfuric acid as precursor reagents. The main idea is to carbonize the saccharose by means of a dehydrolization process with sulfuric acid. Rongalite was used to complex some intermediate carbon structures due its powerful reductor capability.

1gr of saccharose was mixed with 2ml of deionized water in a glass beaker at room temperature until it dissolved, then 0.3ml of polyethyleneimine (PEI, C₂H₅N₂), 0.3ml of rongalite (CH₃NaO₃S), 3ml of sulfuric acid (H₂SO₄) and 0.3ml of ammonium hydroxide (NH₄OH) buffer pH 11 was added to the mixture. At first the solution become darker and further addition of sulfuric acid made the solution completely black.

The material obtained was characterized by FTIR and Raman spectroscopy and SEM-EDS studies.

The samples were characterized by infrared transmission spectra carried out by a Horiba- Jobin-Yvon LabRam HR with a He-Ne laser at 632.8 nm, the chemical reaction give rise to a peak at 1645 cm⁻¹, which is corresponding to a stretching vibration mode of C=C bond, the intensities, positions and widths of the observed peaks are well consistent for the most part in the spectrum, it is inferred that the C=C bond was formed by a dehydration of the saccharose.

The Raman spectra of the sample are show in figure 2, it was carried out by a Perkin Elmer Spectrum two with a UATR module, the spectrum was normalized by the maximum values of G-band peak, the spectrum was collected in the range from 0 to 4000 cm⁻¹, the peaks shown are the so called "G-peak" (for graphite) at 1584 cm⁻¹ and "D-peak" (for disorder) at 1358cm⁻¹. Band peaks characteristics are strongly dependent on structure of the graphitization [3] and the Raman spectra are interpreted by models and theories [4]. The G-peak is due to the phonon mode that allowed by a break-up of selection rules for crystallite with sizes smaller than 100nm (nanocrystalline graphite), the spectra also shows a strong peak in the 2500 cm⁻¹ characteristic of the sp² carbon materials.

The scanning electron microscopy (SEM) were carried out by a FE-SEM JOEL jsm-7800F, images of the formed particles are shown in the figures 3 and 4, the sample has a ordered and homogenous morphology, the surface is covered by small nano-sized grains of about 100nm.

The Elemental composition of the samples has been determined from EDS. The EDS spectrum show the presence of C of 41.88% and 47.36% of oxygen in atomic mass percentages, also a mapping EDS analysis shows a homogenous elemental composition of carbon, oxygen, sulfur, sodium and nitrogen in the sample [5].

References:

- [1] B. McEnaney, in: T.D. Burchell (Ed.), Carbon Materials for Advanced Technologies, (Pergamon, Oxford) 1999 (Chapter 1).
 [2] M. Inagaki, L.R. Radovic, Carbon **40** (2002), p. 2279.
 [3] P. Lespade, R. Al-Jishi, M.S. Dresselhaus, Carbon **20** (1982), p. 427.
 [4] A.C. Ferrari, J. Robertson, Phys. Rev. B **61** (2000) p. 14095.
 [5] The authors acknowledge to CONACyT with project number 255791-INFR-2015.

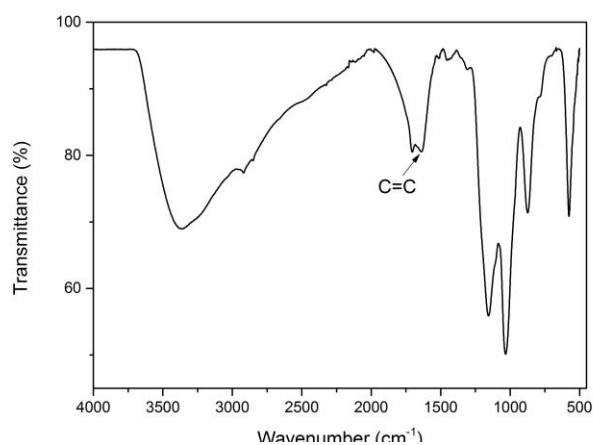


Figure 1. Infrared spectra of the nanostructure carbon Samples.

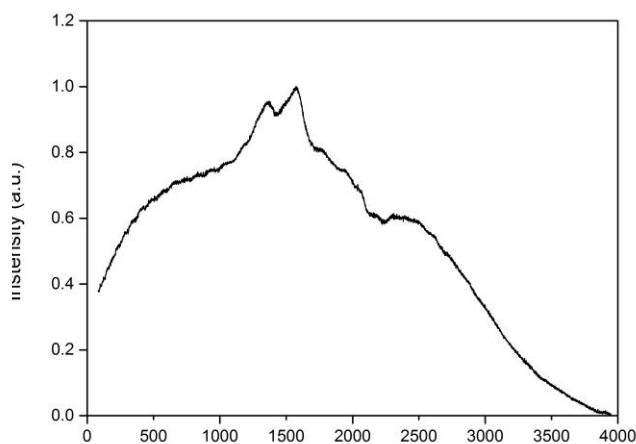


Figure 2. Raman spectra of the samples

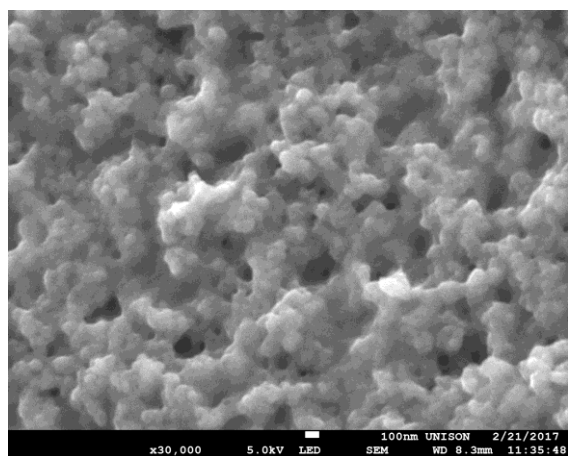


Figure 3. SEM image of the sample with 15000 magnification.

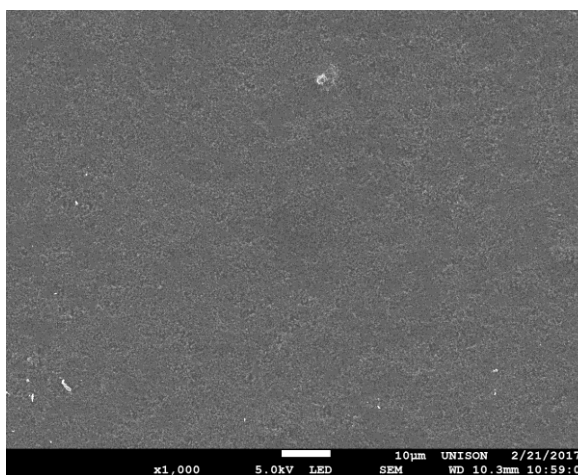


Figure 4. SEM image of the sample with 30000 magnification.