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Structural Analysis of Carbon Nanotubes of Various Diameters Grown by Spray Pyrolysis using Raman Spectroscopy

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Carbon nanotubes of various diameters and length were grown through the alternative spray pyrolysis method [1-2], using propanol, buthanol and cyclohexanol as the carbon source, at temperatures higher than 700°C. In this investigation,we present a comparative analysis of the structures and morphology of the three samples by Raman spectroscopy in order to verify whether it is possible, by using this method, to obtain carbon nanotubes of small diameters at a low cost. The Raman spectra were acquired by the LabRam Horiba HR system using a He- Ne laser at 632.8 nm and 14.2 mW, equipped with a CCD detector column at 75 ° C. The resolution obtained was of approximately 1 cm^{-1}.

According to the literature, this study presents the responses found at different frequency regions: (1) The peaks occurring at a low frequency < 200 cm^{-1} are characteristic only of SWNT assigned to RBM (Radial Breathing Mode), and their frequency depends essentially on the diameter of the nanotubes [3]. (2) The peak at 1340 cm^{-1} is assigned to the poorly-organized residual graphite. In this region the peak is related to the so-called D band (Band Disorder) [4-5].(3) The frequencies between 1500 cm^{-1} and 1600 cm^{-1} are related to the G band and are highly characteristic of single nanotubes and multiple wall, These frequencies correspond to a splitting of the graphite extension mode (stretching mode) [3-5]. (4) A second order mode vibration, between 2450 cm^{-1} and 2650 cm^{-1} , is assigned to the first on-tone mode and to the D mode, sometimes called G´ [3-5].

Figure 1(a) shows the Raman spectrum of the propanol sample at high frequencies, from 500 to 3000 cm^{-1}. The characteristic D band and G band peaks are observed. Band D is at 1321 cm^{-1} , and it is more intense than the G band at 1582 cm^{-1} given. This indicates the presence of poorly organized graphite and the presence of possible defects in carbon nanotubes. The presence of the band G` 2635 $cm^{-1}$, corresponds to second order dispersal processes [6]. The fact that both bands, D and G`, have high intensity peaks indicates structural imperfections in the sample. Figure 1(b) shows a Raman spectrum of low frequency region. Three peaks around 161 cm^{-1} , 223 cm^{-1} and 267cm^{-1} are featured. The first two peaks indicate the presence of single-walled nanotubes with different chirality, and the third peak nearby 267 cm^{-1}, corresponds to the response of the quartz substrate used for the growth of nanotubes.

Figure 2(a) displays the Raman spectra of the butanol sample in the high frequency region. We can observe that Band D is at 1340 cm -1 , and Band G is given at 1587 cm^{-1} and 2663 cm^{-1}. Bands G` and D , as propanol , are stronger than Band G, thus indicating a high density of structural imperfections, possibly poorly-organized graphite. In Figure 2(b), the low frequency region, we can observe peaks at 154 cm^{-1} and 229 cm^{-1} and 294cm^{-1}. By considerating only the first two peaks, the possible presence of single-walled nanotubes of various types of chirality can be confirmed. The peak around 294 cm^{-1} is related to the substrate.The cyclohexanol sample, where carbon nanotubes were grown at temperatures of 850 ^{0}C, can be observed in Figure 3(a). Here, Band D is located at 1332 cm^{-1}, Band G at 1597 cm^{-1}

and Band G` at 2657 cm $^{-1}$. In this sample, the intensity of Band D is lower, indicating that the sample has a low density of structural imperfections as well as poorly-organized graphite.Figure 3(b). Shows low energies where two predominant curves around 147 cm^{-1} and 186 cm^{-1} are observed, indicating two possible types of single-walled nanotubes with different diameters. The other peaks present are related to the quartz substrate.

In conclusion; carbon nanotubes were grown at a temperature of 700°C for propanol and buthanol. In the case Cyclohexanol, they were grown at 850°C. Raman spectroscopy reveals that the three samples containing the presence of possible carbon nanotubes of small diameters answer to regions of very low frequency. However, all samples with structural defects by analysis of their responses occurred at high energies, which challenges us to continue testing to get better quality nanotubes and small diameters at a low cost.

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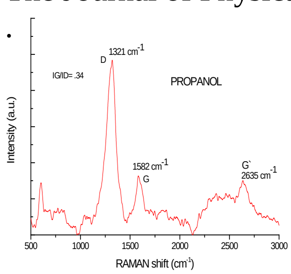
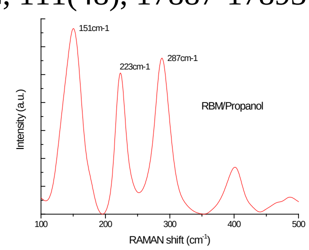
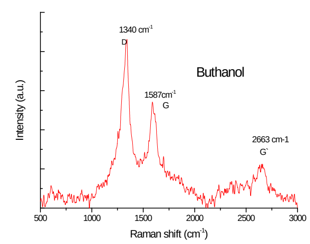
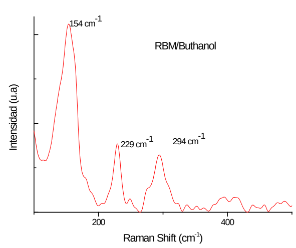
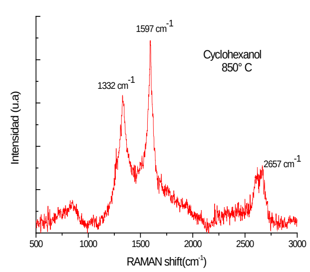


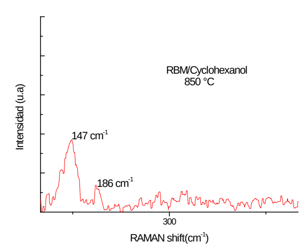




Figures 1a, Raman spectrum of the propanol sample at high energies.. **1b** Raman spectrum of the propanol sample at low energies.**2a,** Raman spectrum of the buthanol sample at high energies.







Figures 2 b Raman spectrum of the buthanol sample at low energies.**3a** Raman spectrum of the cyclohexanol sample at high energies.**3b.** Raman spectrum of the cyclohexanol sample at low energies.