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Characterization of Few Walled Carbon Nanotubes using Aliphatic Alcohols as Carbon Source

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We present an alternative process that can lead to lower the manufacturing cost of few walls CNTs by means of the spray pyrolysis technique. For this purpose, ferrocene is utilized as a catalyst and aliphatic alcohols (methanol, ethanol, propanol, butanol) as the carbon source. The characterization of the produced CNTs was performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Some reports have mentioned the use of alcohols as carbon source for CNTs synthesis and have completed a study of the product regarding the effect of the alcohol carbon length, type of catalysts used and/or changes by the CVD method [1-4]. Other authors mention the use of alcohols ranging from methanol to decanol, where single walled nanotubes (SWNT) were obtained from alcohols of one to six carbon atoms and multiwall nanotubes (MWNT) with longer chain alcohols, stating that carbon chains in alcohol molecules influence the crystallinity of the product [5-7]. Nevertheless, the reported synthesis implies a low rate production method, since it takes around three hours to produce quantities between 200 to 400 mg of nanotubes.

The alternative spray pyrolysis method used in this work consists in the introduction of the precursor into a stainless steel pre-heater maintained at constant temperature with the purpose of changing the liquid precursors to their vapor phases, before entering the reaction chamber where the CNTs are grown up in 25 minutes. Using this method we anticipate the establishment of a new base that allows producing low diameter CNTs close to the reported SWCNTs with a low cost advantage. Morphology and microstructural characterization of the CNTs were performed by scanning electron microscopy (SEM) in a JSM-7401F instrument operated at 3-5 kV and by high resolution transmission electron microscopy (HRTEM) in a JEOL JEM-2100FS with beam Cs-corrector, operated at 200 kV, with a spatial resolution close to 0.13 nm.

Figure 1 shows SEM images that reveal the variation of morphology for the CNTs as a function of the alcohol used as carbon source. Average values of the size parameters were obtained by measuring length and diameter of the most representative nanotubes from each sample, and results are shown in Table 1. It is observed that the average diameter varies inversely to the number of carbon atoms contained in the precursor molecule of the alcohol used and the average length is directly proportional to the number of carbon atoms. Analysis using energy dispersive X-ray spectrometry (EDS) shows the presence of iron nanoparticles. The quantity of nanotubes collected was, on the average, 0.65 g for methanol, 0.30 g for ethanol and 0.35 g for propanol and butanol. The number of layers that constitute the nanotubes walls, are observed from the HRTEM micrographs, shown in Figure 2, where it can be seen how the iron nanoparticles (identified with an arrow) promotes the formation of graphite walls that constitute the CNTs. The methanol sample (Fig.2-a), shows that the number of walls that conforms these CNTs is typically of the order of 35. In the ethanol case (Fig. 2-b), the iron nanoparticles are bigger than the nanoparticles in the other precursors and the number of walls diminishes to about 10. Figs. 2c-d shows HRTEM micrographs from propanol- and butanol-based CNT, respectively. It is observed that the average numbers of nanotubes walls are similar and about 6 for both propanol and butanol.

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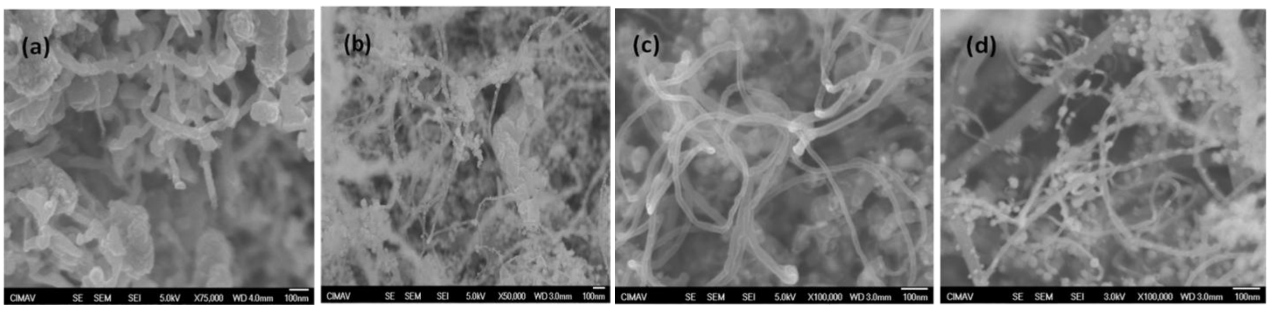

Figure 1. SEM images of carbon nanotubes obtained under special synthesis conditions using (a) Methanol/ferrocene, (b) Ethanol/ferrocene, (c) Propanol/ferrocene and (d) butanol/ferrocene**.**

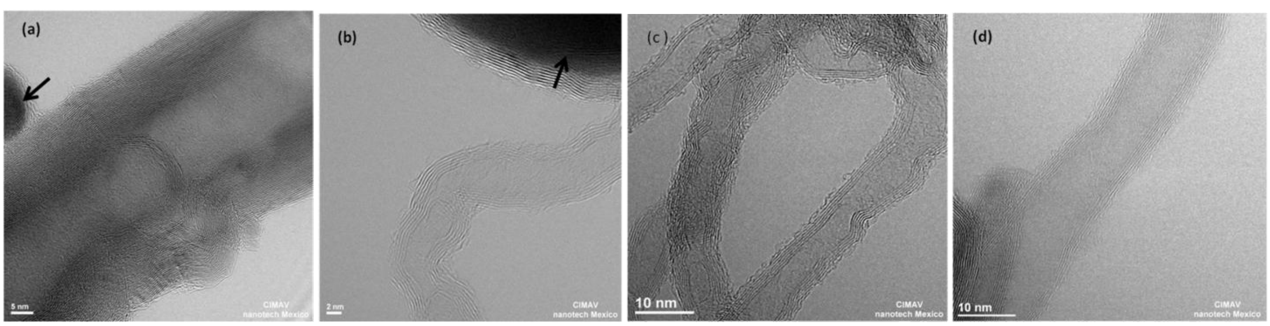

Figure 2. TEM images of CNT`s obtained under special synthesis conditions using (a) Methanol/ferrocene,(b) Ethanol/ferrocene, (c) Propanol/ferrocene and (d) butanol/ferrocene.(Arrows indicate iron nanoparticles and carbon encapsulates**.**

Table. 1. Average external diameters, lengths and number of walled of carbon nanotubes.

ALCOHOL	AVERAGE EXTERNAL DIAMETER (nm)	AVERAGE LENGTH (nm)	AVERAGE NUMBER OF WALLS
Methanol (CH_4O)	24	0.5	35
Ethanol (C_2H_6O)	18	1.2	10
Propanol (C_3H_8O)	15	2.0	8
Butanol ($C_4H_{10}O$)	16	2.2	6

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