Purification and Characterization of Single-Wall Carbon Nanotubes Synthesized by Pulsed Laser Vaporization.

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Currently, there is a need for ultra-high purity single-wall carbon nanotubes (SWNTs) for a variety of products, such as multi-functional polymer composites, and applications, such as hydrogen storage. However, the existing purification methods still produce a material that contains residual metal catalyst and amorphous and graphitic carbon. The goal of these studies was to optimize the purification of SWNTs prepared by pulsed laser vaporization to minimize the impurities and maximize the yield of SWNTs. In our studies, SWNTs were synthesized by laser vaporization of a Dylon target containing 1atomic % Ni and Co at 1150 °C under a flow of argon. To optimize the production of SWNTs, the reaction parameters including laser power, pulse length, temperature, gas flow, and target material, was varied. The crude SWNTs were characterized by SEM/EDX, TEM, TGA, Raman, and IR spectroscopy, and they were found to contain residual metal catalyst (up to 12 wt%) and amorphous carbon. The SWNTs were purified by refluxing in nitric acid followed by air oxidation at elevated temperatures and washing in concentrated acid. The reaction parameters, such as acid concentration, reflux time, thermal oxidation time and temperature, have been investigated to optimize the purity and yield of SWNTs. In general, it was found that multiple oxidation/acid washing steps were better than a single oxidation step since the residual metal can catalyze the oxidation of the nanotubes. To obtain the best purity and yield, it is beneficial to optimize the oxidation time and temperature for each sample by TGA before scaling up the process. Currently, this method produces 8±1 wt% SWNTs that contained a low content of amorphous carbon and metal (≤0.1 wt%).

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