Supplementary Information

Fullerenes as corks for the containment of materials inside open-ended single-walled carbon nanotubes

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Purification and filling of CVD SWNTs

Carbon nanotubes (CNTs) prepared by chemical vapour deposition were supplied by Thomas Swan & Co. Ltd. The samples contain single-walled carbon nanotubes (SWNTs) and a fraction of double-walled carbon nanotubes (DWNTs) both of which performed in the same way under the reported experimental conditions. Therefore we refer to the sample as SWNTs. As-received SWNTs were opened and purified using steam[1] at 900°C for 4 h. The sample was then treated with HCl to remove metal particles, now free of graphitic coating,[2] rinsed with water and dried at 100 °C. A further treatment in air[3] (570 °C, 15 min) was done to improve the opening yield of SWNTs. These opened SWNTs were then filled with uranyl acetate, by stirring 10 mg of opened SWNTs with an aqueous solution of uranyl acetate (170 mg uranyl acetate in 10 mL distilled water) at 70°C for 24 h. The solution was next filtered, and the solid residue (thick texture) on the filter membrane was sonicated briefly with 100 mL of distilled water and filtered again. The sample was then dried at 100 °C overnight.

In order to fill fullerenes into the uranyl acetate filled SWNTs, C_{60} crystallites (1 mg) were first sonicated in ethanol (10 mL) for 3 min. Uranyl acetate@SWNTs (1 mg) were then added to this fullerene solution which was sonicated for an extra minute. The mixture was left standing in the ethanol solution for 48 h at room temperature.[4] The solid powder was then removed from the bottom of the ethanol solution and allowed to dry at room temperature. To remove the unfilled uranyl acetate, the sample was sonicated for 5 min in 100 mL of 1 M HCl and stirred for 24 h. The sample was then filtered. This washing step was repeated four times.

Purification and filling of arc-discharged SWNTs

SWNTs prepared by arc-discharge using a Ni-Y catalyst[5] were steam purified[1] at 900°C for 2 h. The sample was then treated with HCl to remove metal particles free of graphitic coating (after steam),[2] rinsed with water and dried in air at 100 °C. SWNTs (0.035 g) were ground together with KI (0.7 g) using an agate mortar and pestle. The mixture was then sealed in a silica ampoule under vacuum. The ampoule was heated to 781 °C at a rate of 3 °C/min., maintained at this temperature for 4 h and then cooled down to room temperature (3 °C/min.). The sample was washed by distilled water and filtered. An opening step was needed in this case for the incorporation of C_{60}. The SWNTs tips were removed by heating the sample in air at 420 °C for 20 min.

The filling of C_{60} (5 mg) into the opened KI@SWNTs (5 mg) was carried out in a sealed quartz ampoule under vacuum at 400 °C for 48 h.[6] The mixture was then dispersed in hexane and filtered to remove the excess of C_{60}. The external KI was removed by sonicating the sample in 100 mL of distilled water for 5 min and stirring during 24 h. The sample was recovered by filtration. The washing step was repeated four times.

High resolution electron microscopy (HRTEM)

HRTEM images were taken in a JEOL JEM-4000EX high resolution electron microscope (point resolution, 0.16 nm). Samples for HRTEM observation were ground and dispersed in ethanol and placed dropwise onto a holey carbon support grid**.**

References

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