Supplementary Information for

Steaming multiwalled carbon nanotubes via acid vapor for controllable

nanoengineerings and the fabrication of carbon nanoflutes

Jun Ming,^{a,b,c} Yingqiang Wu,^{a,b,c} Yancun Yu,^{a,b} Fengyu Zhao^{a, b}*

^aState Key Laboratory of Electroanalytical Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, P. R. China; ^bLaboratory of Green Chemistry and Process, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, P. R. China; ^cGraduate University of the Chinese Academy of Sciences, Beijing100049, P. R. China.

Materials

The multiwalled carbon nanotubes (MWCNTs) (length 5-15 μ m, diameter 40-60 nm, purity >98%) were purchased from Shenzhen Nanotech Port Co., Ltd. The concentrated HNO₃ (65~68 wt.%) and HCl (36~38 wt.%) were purchased from Sinopharm Chemical Reagent Beijing Co. Ltd and used directly without any dilution. A sample of MWCNTs@amorphous carbon (50.68 wt.%) & metal (M = Fe, Ni, Co, total 16.97 wt.%) (Fig. S3A), synthesized by coating MWCNTs with a layer of amorphous carbon^{S1} and loading the metal nanoparticles (Fe, Co, Ni)^{S2}, was used to examine the capability of the present steaming strategy in removing the impurities of metal and/or amorphous carbon.

Experimental procedure

A typical steaming procedure for surface functionalization of MWCNTs is as follows: 0.3 g pristine MWCNTs (p-MWCNTs) were loaded on the porous SiO₂ griddle of glass steamer (Fig. S1A) and then placed into a 50 mL Teflon-vessel (Fig. S1B), at the bottom of which 0.15-3 mL HNO₃ was added previously. Subsequently, the Teflon vessel was sealed in the autoclave and then moved to the oven for steaming at the temperature of 120-200 °C within 5 h (Fig. S1, C to E). After the steaming treatment, cooled down the autoclave to the room temperature and took the steamer out of the Teflon vessel into a clean beaker, in which washed the functionalized MWCNTs (f-MWCNTs) with distilled water and ethanol by *in-situ* filtration (Fig. S1F). Finally, the steamer was transferred to the oven for drying (60 °C) and the formed solid black cakes (f-MWCNTs, inset of Fig. S1F) could be collected easily and completely. Normal surface functionalization, short-cut, end-open, production of carbon nanoflutes, removal of amorphous carbon and some metal catalysts that could not react with HCl, should be treated by HNO₃. Alternatively, the HCl was used for removing most transitional metal impurities or catalysts.

References:

- s1 Y. Wan, Y. L. Min and S. H. Yu, *Langmuir* 2008, 24, 5024-5028.
- s2 X. Meng, H. Cheng, Y. Akiyama, Y. Hao, W. Qiao, Y. Yu, F. Zhao, S. Fujita and M. Arai, J. Catal. 2009, 264, 1-10.



Fig. S1 (A) The photograph of glass steamer and the placement of MWCNTs. (B) The Teflon vessel (50 mL). (C) The stainless steel autoclave with a cover padded by Teflon. (D) The situation of equipments (E) The schematic procedure of the steaming process. (F) The washing and separation of f-MWCNTs through filtration *in-situ* in the glass steamer.

Supplementary Material (ESI) for Chemical Communications This journal is (c) The Royal Society of Chemistry 2011



Fig. S2 The dispersion ability of p-MWCNTs and f-MWCNTs with different weight losses in water (A) and in organic solvents (B), ethylene glycol (EG).

Supplementary Material (ESI) for Chemical Communications This journal is (c) The Royal Society of Chemistry 2011



Fig. S3 (A) MWCNTs@amorphous carbon (50.68 wt.%) & metal (M = Fe, Ni, Co, total 16.97 wt.%), (B) MWCNTs@amorphous carbon obtained after selectively removed the metal impurities by steaming with HCl, (C) MWCNTs obtained after simultaneously removed the metal impurities and amorphous carbon by streaming with HNO₃. 1-amorphous carbon, 2-the multi-walls of MWCNTs, 3-the hollow tubes of CNTs.



Fig. S4 (a) Nitrogen adsorption isotherms of the p-MWCNTs and f-MWCNTs with different weight losses. Inset figure is the changing relations between the weight losses and the specific surface area of f-MWCNTs. (b) The pore size distribution of p-MWCNTs and f-MWCNTs. Upper inset figure is the changing relations between the weight loss and the pore volume of f-MWCNTs, below inset figures are TEM mages of p-MWCNTs and f-MWCNTs with varied weight losses.