Microwave Synthesis of Nanocarbons from Conducting Polymers Xinyu Zhang, Alan G. MacDiarmid and Sanjeev K. Manohar*

Alan G. MacDiarmid Center for Innovation, Department of Chemistry, The University of Texas at Dallas, Richardson, TX 75080

Synthesis of polypyrrole nanospheres:

To 60 ml of a stirred 0.14M solution of pyrrole in aq. 1.0M HCl, 40 ml of a 0.04M solution of the oxidant ammonium peroxydisulfate, also in aq. 1.0M HCl was added. After 20 min, the resulting black precipitate of polypyrrole.Cl was suction filtered, washed with copious amounts of aq. 1.0M HCl, and dried under dynamic vacuum at 80° C for 12h. The yield of polypyrrole.Cl powder obtained was ~300 mg.

Synthesis of polypyrrole nanofibers:

To 60 ml of a stirred 0.14M solution of pyrrole in aq. 1.0M HCl, was added \sim 1-4 mg of nanofibers of the seed template. After 1 min, 40 ml of a 0.04M solution of the oxidant ammonium peroxydisulfate, also in aq. 1.0M HCl was added. After 20 min, the resulting black-brown precipitate of polypyrrole.Cl was suction filtered, washed with copious amounts of aq. 1.0M HCl, and dried under dynamic vacuum at 80°C for 12h. The yield of polypyrrole.Cl powder obtained was \sim 300 mg.

Synthesis of polypyrrole nanotubes:

Polypyrrole nanotubes were synthesized by V_2O_5 -templating as follows. A mixture of ethanol (50 ml) and V_2O_5 sol-gel (2.0 ml), was magnetically stirred for 12 h and pyrrole monomer (14.9 mmol) was added followed by anhydrous FeCl₃ oxidant (10 mmol) was added to initiate the polymerization. The solution immediately turned dark and after 1 h the black precipitate of polypyrrole nanotubes having 10-15 nm diameter V_2O_5 pores was suction filtered and suspended in aq. 1.0M HCl (100 ml) for 2h to leach out the V_2O_5 from the pore. Suction filtration followed by drying under dynamic vacuum at 80°C for 12 h yielded ~50 mg of polypyrrole nanotubes.

Synthesis of nanocarbons:

Nanocarbons are synthesized by microwave heating the conducting polymers polypyrrole, poly(3,4-ethylenedioxythiophene) (PEDOT) etc. The procedure is as follows: accurately weighed amount (20-50 mg) of the nanostructured polypyrrole is placed in a glass vial. For large scale synthesis, multiple vials (6-12) could be put into the microwave oven at the same time. The microwave oven is adjusted to 800W for the output power. As soon as the microwave radiation starts, there is extensive sparking along the polypyrrole or PEDOT polymer surfaces accompanied by a rapid increase in temperature. The samples are heated for 3 min continuously. If the microwave heating continues beyond 3 min, the glass vial becomes red hot and even melts. After the heating procedure, let the sample cool down to room temperature and weigh it. The yield of nanocarbons from conducting polymers is 30–40%.

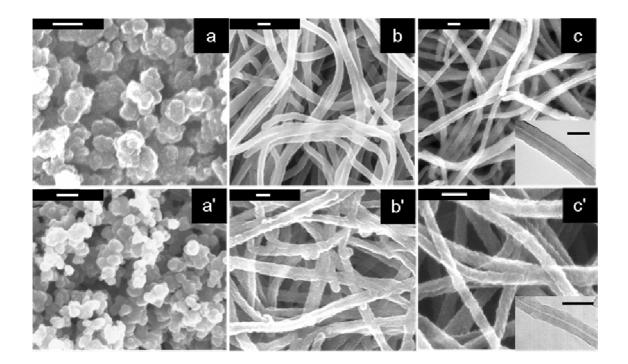


Fig. S1. Enlarged image of Figure 1 in manuscript

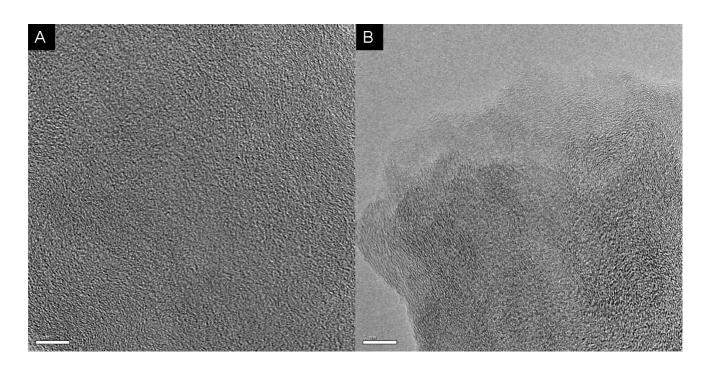


Fig. S2. Representative HRTEM images of polypyrrole.Cl nanofibers before (A) and after (B) microwave heating for 3 min. Scale: 5 nm.

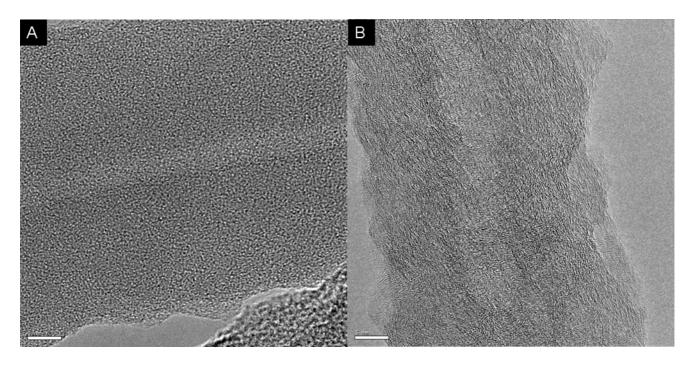


Fig. S3. Representative HRTEM images of polypyrrole.Cl nanotubes before (A) and after (B) microwave heating for 3 min. Scale: 5 nm.