



Chiral carbonaceous nanotubes prepared by carbonization of chiral organic self-assemblies

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ABSTRACT

Single-handed helical tantalum pentoxide nanotubes with inner chiral organic self-assemblies were prepared through a supramolecular templating approach. After carbonization and removal of tantalum pentoxide, single-handed twisted carbonaceous nanotubes were obtained. The handedness of the carbonaceous nanotubes followed that of the tantalum pentoxide nanotubes. The carbonaceous nanotubes were formed due to the adsorption of the organic self-assemblies on the inner surfaces of the tantalum pentoxide nanotubes and the shrinkage of organic self-assemblies during the drying process and subsequently carbonization. The X-ray diffraction and Raman spectroscopy characterizations indicated that the carbon was amorphous. The circular dichroism spectra indicated that the twisted carbonaceous nanotubes exhibited optical activity. The elemental analysis showed that these carbonaceous nanotubes were N-doped. These N-doped carbonaceous materials might find applications as catalysts and chirality sensors.

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1. Introduction

Nanoscience-control of matter has boosted the development of carbon materials [1]. For example, ordered mesoporous carbon materials have attracted more and more interest owing to their high surface area and uniform pore channel, which has potential application in catalysis, drug release and advanced electronic materials [2–4]. Helical carbon nanomaterial is another kind of interesting carbon materials because of their diverse applications in electromagnetic waves absorption, hydrogen storage and field emission materials [5–7]. They have been successfully prepared through the chemical vapor deposition method [5,6]. For controlling the handedness of the carbonaceous nanotubes, the method of carbonization of polymeric nanotubes was developed.

Single-handed helical organopolymeric nanotubes, such as polyaniline [8], polypyrrole [8,9], 3-aminophenol-formaldehyde resin [10], were prepared using the self-assemblies of chiral amphiphilic molecules as templates. Single-handed carbonaceous nanotubes were obtained after carbonization of these organopolymeric nanotubes [9–11]. Single-handed helical hybrid silica nanotubes can be also used as the starting materials [12]. After carbonization and removal of silica, carbonaceous nanostructures

were obtained. Recently, another method of carbonization of organic compounds within silica nanotubes was developed. Single-handed helical carbonaceous nanotubes containing twisted carbonaceous nanoribbons were obtained [13]. Helical metal oxide nanotubes have been prepared by template method [14,15]. Herein, single-handed twisted tantalum pentoxide nanotubes containing inner organic self-assemblies were prepared firstly via a sol-gel duplication method. After carbonization at 600 °C in Ar and removal of tantalum pentoxide, left-handed twisted carbonaceous nanotubes with about 6 wt% N content was obtained successfully.

2. Experimental section

2.1. Materials

Aqueous hydrofluoric acid solution and ammonium hydroxide (25.0 wt%) were purchased from Sinopharm Chemical Reagent Co. Tantalum (V) ethoxide was purchased from Alfa Aesar. The synthesis and characterization of the LMWGs, *LL*- and *DD*-12Val5PyClO₄, have been reported previously (Fig. S1) [14].

2.2. Preparation of single-handed helical carbonaceous nanotubes

LL-(*DD*) 12Val5PyClO₄ (31.4 mg, 0.034 mmol) was dissolved in absolute ethanol (0.7 mL). Then tantalum(V) ethoxide (0.12 mL, 0.

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46 mmol) and concentrated ammonium hydroxide (5.0 μL , 25.0 wt %) were added to the mixture under stirring. An opaque gel was formed after the reaction mixture was cooled to room temperature. The gel was dried in an open test tube. The obtained tantalum oxide nanotubes with inner LMWGs were carbonization at 600 $^{\circ}\text{C}$ for 3.0 h in Ar with heating rate of 3.0 $^{\circ}\text{C min}^{-1}$. To remove the tantalum pentoxide, the obtained samples were immersed in aqueous HF (5.0 wt%) for 24 h and then washed with deionized water. The obtained left- and right-handed twisted carbonaceous nanotubes are named as *M-C* and *P-C*.

3. Results and discussion

LL- and *DD-*12Val5PyClO₄ can self-assemble into right- and left-handed helical bundles in ethanol, respectively. Right- and left-handed helical nanotubes with inner template were obtained using *LL-* and *DD-*12Val5PyClO₄ assemblies as template, respectively (Fig. 1) [14]. The TEM images indicated a tubular structure (Fig. 1c and d). The diameters and helical pitch of the nanotubes were in the range of 100–500 and 400–800 nm, respectively. The lengths of tantalum pentoxide nanotubes are several micrometers. Some thinner straight tantalum pentoxide nanotubes (approximately 50–100 nm) were also identified in the FE-SEM images (Fig. 1a and b).

After carbonization, the morphologies of tantalum oxide/carbon did not change (Fig. S2). After removal of the tantalum pentoxide, the carbonaceous nanotubes were obtained. The composition of *M-C* was analyzed by EDX (Fig. S3). The result shows that the tantalum content is 1.25 wt%, which indicate that tantalum pentoxide

were mostly removed. The FE-SEM and TEM images of *P-C* and *M-C* are shown in the Fig. 2. The carbonaceous nanotubes successfully maintained the structures of tantalum pentoxide nanotubes, except significant shrinkage. The width and helical pitch of these twisted carbonaceous nanotubes were 100–170 and 150–500 nm, respectively. Some straight carbonaceous nanotubes were also identified in the FE-SEM images. The TEM images indicated that the wall thickness of nanotubes were about 10 nm. The elemental analysis result of *M-C* indicated that carbon and nitrogen contents were 87.31 wt% and 6.01 wt%. XPS measurements showed the existence of pyridine-like, pyrrolic-like, graphitic-like nitrogens and pyridine-N-oxide in the carbonaceous nanotubes (Fig. S4). The intentional incorporation of nitrogen atoms into the carbonaceous nanostructures could effectively modify its electronic and chemical properties, which endowed it great potential to be an efficient anode material of rechargeable lithium ion batteries [16].

Single-handed helical tantalum pentoxide nanotubes exhibited optical activity, as shown in Fig. S5. The left- and right-handed helical tantalum pentoxide nanotubes showed opposite CD signals. The CD and UV-vis spectra of left- and right-handed twisted carbonaceous nanotubes are shown in the Fig. 3. Broad UV-vis absorption bands at $\lambda = 220$ and 700 nm can be observed. Although the *M-C* and *P-C* exhibited not exactly opposite CD signal, the samples exhibit optical activity. The $\lambda_{0=0}$ value were presented at $\lambda = 604$ nm, larger than that of carbonaceous nanotubes containing twisted carbonaceous nanoribbons, the wavelength shift is due to the enlargement of aromatic rings [13]. For *M-C*, the first signal was negative and one broad positive CD signal centered at 365 nm. The CD signals were proposed to originate from the electron

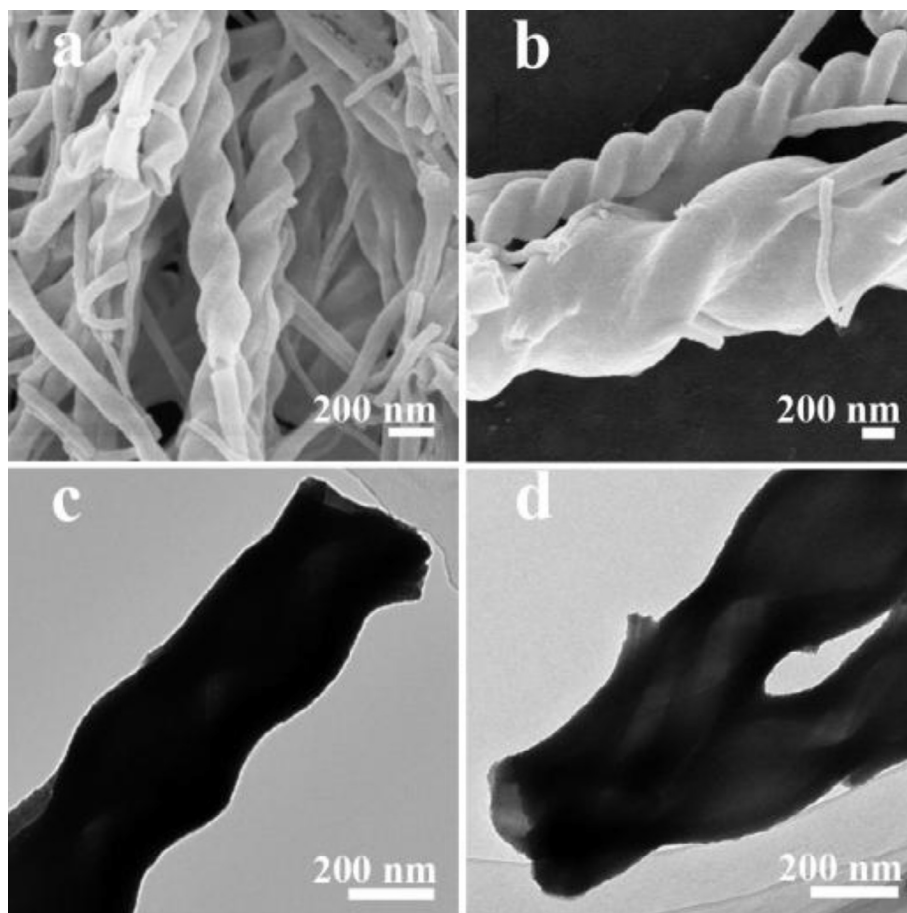


Fig. 1. FE-SEM (a, b) and TEM (c, d) images of right-handed (a, c) and left-handed helical tantalum pentoxide nanotubes with inner template (b, d).

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