

Sonoelectrochemical Synthesis of CdSe Nanotubes

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Experimental details:

Synthesis of CdSe Nanotubes: All chemicals were used as received without further purification. The room-temperature synthesis of the CdSe nanotubes is based on constant current electrodeposition accompanied by continuous sonication in an aqueous solution that contains cadmium chloride ($\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$), sodium nitrilotriacetate ($\text{N}(\text{CH}_2\text{COONa})_3$, NTA), sodium selenite (Na_2SeO_3) and polyvinylpyrrolidone (PVP). A VCX-750 ultrasonic processor (Ti horn, 1.13 cm in diameter, 20 kHz, Sonics & Materials) was used as the ultrasound source and a CHI6301B electrochemical workstation acted as the current source.

In a typical procedure, $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ (0.68 g) and PVP (1.0 g) were dissolved in 50 ml of NTA (0.1molL^{-1}) solution, and then Na_2SeO_3 (0.26g) was added. The solution was sonicated with current densities in the range of $60\sim 80\text{ mA}\cdot\text{cm}^{-2}$, ultrasound intensities of approximately 25%, and a reaction time of 2 h. After the reaction, the resulting brown black solution was purified by centrifugation, and the precipitate was

washed sequentially with distilled water and ethanol for several times and then dried in air for characterizations.

Characterization: Wide-angle powder X-ray diffraction (XRD) patterns were obtained with a Philips X'pert Pro X-ray diffractometer (CuK α radiation, $\lambda=0.15418\text{nm}$). Scanning electron microscopic (SEM) images were taken on a HITACHI-S4800 scanning electron microscope combined with energy dispersive spectroscopy (EDS, INCA 300). The transmission electron microscopic measurements were carried out on a JEOL 200CX transmission electron microscope using an accelerating voltage of 200 kV. High resolution transmission electron microscopic (HRTEM) images were obtained on a JEOL 4000EX electron microscope using an accelerating voltage of 400 kV.

The electrochemical measurement for ECL was carried out on a CHI 812 electrochemical working station (Shanghai CH Instruments Co., China) using a three-electrode system. The electrodes were a carbon paste working electrode modified with CdSe nanotubes, a saturated calomel reference electrode (SCE), and a Pt counter electrode. Carbon paste electrodes were prepared according to the report.¹ The ECL emission was detected with a Model MPI-A Electrochemiluminescence Analyzer (Xi'An Remax Electronic Science & Technology Co. Ltd., Xi'An, China) at room temperature. The spectral width of the photomultiplier tube (PMT) was 200-800 nm and the voltage of the PMT was set at 800 V in the process of detection. The electrode potential was cycled between 0.1 and -1.2 V at a scan rate of 100 mV s⁻¹

The XRD pattern of CdSe nanotubes

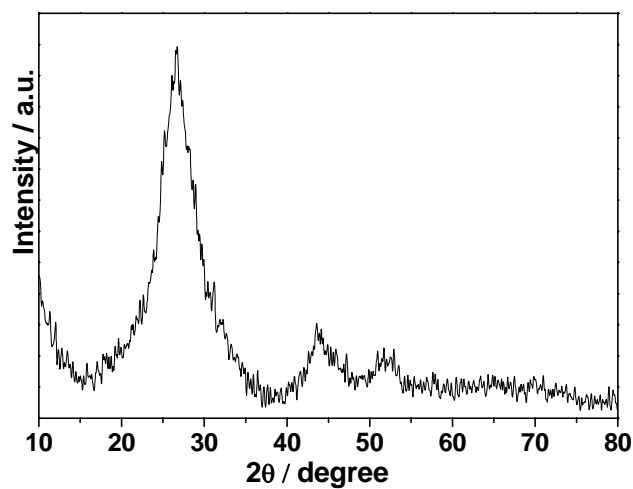


Fig. s1 The XRD pattern of CdSe nanotubes.

The controlled experiments

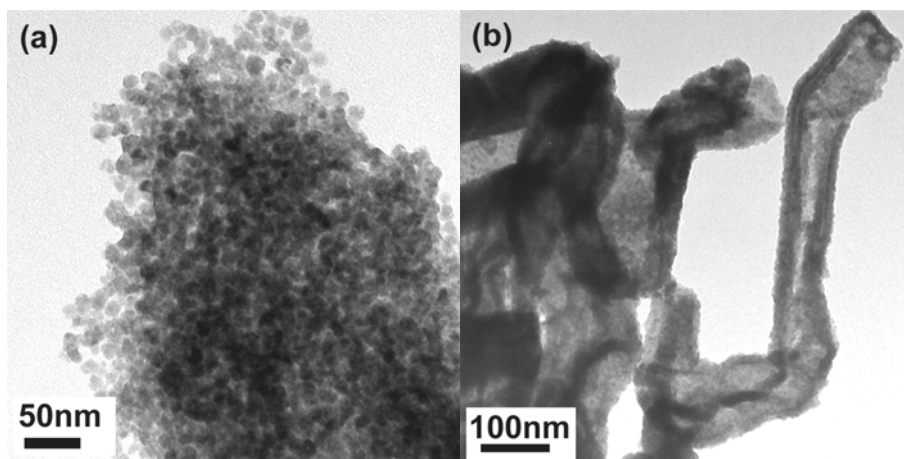


Fig. s2 TEM images of CdSe products synthesized at other conditions. (a) Reduced by N_2H_4 . (b) Sonoelectrochemical synthesis in the absence of PVP.

The ECL measurement

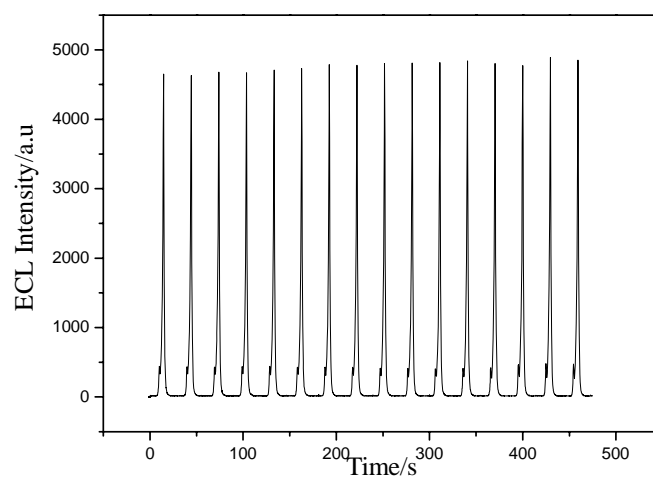


Fig. 3s ECL emission from the CdSe nanotubes in pH 7.4 PBS containing 0.1 M KCl and 0.1M $K_2S_2O_8$ under continuous cyclic voltammetry for 16 cycles. Cycled between 0.1 and -1.2 V at a scan rate of $100 \text{ mV}\cdot\text{s}^{-1}$.

Supplementary references

- 1 T. Ren, J. Xu, Y. Tu, S. Xu and J. J. Zhu, *Electrochem. Comm.*, 2005, **7**, 5; J. J. Miao, T. Ren, L. Dong, J. J. Zhu and H. Y. Chen, *Small*, 2005, **1**, 802.