

Microwave-assisted ionothermal synthesis of SnSe_x nanodots: A facile precursor approach towards SnSe_2 nanodots/graphene nanocomposite

Cheng-Feng Du,^{a,b} Jian-Rong Li,^a and Xiao-Ying Huang^{*a}

^a State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, P. R. China. Fax: (+86) 591-63173145

^b University of Chinese Academy of Sciences, Beijing, 100049, People's Republic of China.

E-mail: xyhuang@fjirsm.ac.cn

Supporting Information

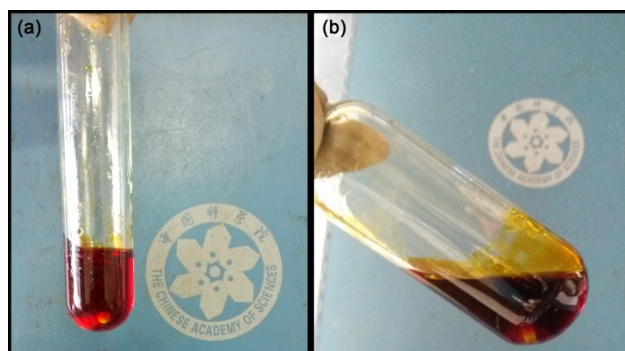


Fig. S1 The as-prepared SnSe_x NDs precursors with different reacting magnitudes. (a) 2 mmol reaction in a 10 mL vial. (b) The scale up reaction of 5 mmol in a 25 mL vial.

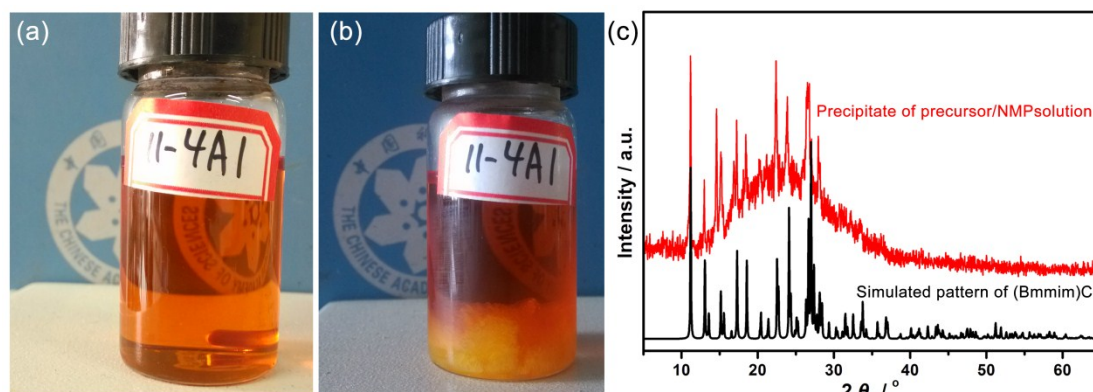


Fig. S2 Photographs of the fresh SnSe_x NDs precursor/NMP solution (a) and chilled solution (b). (c) PXRD patterns of the precipitate obtained from the chilled solution and the simulated pattern of (Bmmim)Cl.

Thermalgravimetric analysis was performed for the $\text{SnSe}_x \text{NDs@Bmmim@GO}$ nanocomposites. The TGA curve in Fig. 3a displayed three steps of weight losses. The first weight loss of around 18.4% was in the range of 100–200 °C, which can be assigned to the evaporation of residual NMP, H_2O and release of CO_2 . The second weight loss of around 12.7 % in the range of 270–350 °C was in accordance with the thermally decomposition of $[\text{Bmmim}]^+$. The third weight loss began at about 370 °C, which could be assigned to the elimination of excess Se during the phase transformation from SnSe_2 to SnSe . The TGA profiles of the pristine nanocomposite, simplex $\text{SnSe}_x \text{NDs}$ precursor and GO powder were depicted in Fig. S3b for comparison.

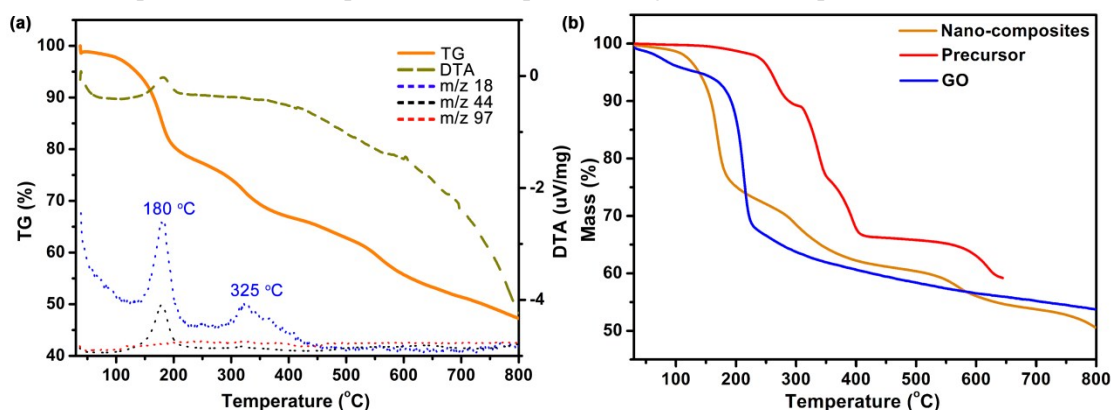


Fig. S3 (a) TG-MS profile of $\text{SnSe}_x \text{NDs@Bmmim@GO}$ composite at a heating rate of $5\text{ }^\circ\text{C min}^{-1}$ in a N_2 atmosphere from 30 to 800 °C. (b) TGA profiles of the pristine nanocomposite, simplex SnSe_2 precursor and GO powder.

As illustrated in Fig. S4a, the PXRD pattern of pristine NDs@Bmmim@GO nanocomposite shows three broad diffraction peaks at around 9, 27 and 47°, which is similar to that of the simplex $\text{SnSe}_x \text{NDs@Bmmim}$ sample. Interestingly, no characteristic peaks of SnSe_2 could be observed from the PXRD pattern, probably due to the formation of new selenidostannate phases. In the PXRD patterns of product after annealing at 300 °C for 2 hours (e.g. NG-3 in Fig. S4A), all the peaks can be readily indexed to the hexagonal SnSe_2 phase (JCPDS card No. 89-3197), indicating high phase purity. When increasing the annealing temperature, phase transformation from SnSe_2 to SnSe can be observed; finally, pure phase of SnSe (JCPDS card No. 48-1224) was obtained (Fig. S4b).

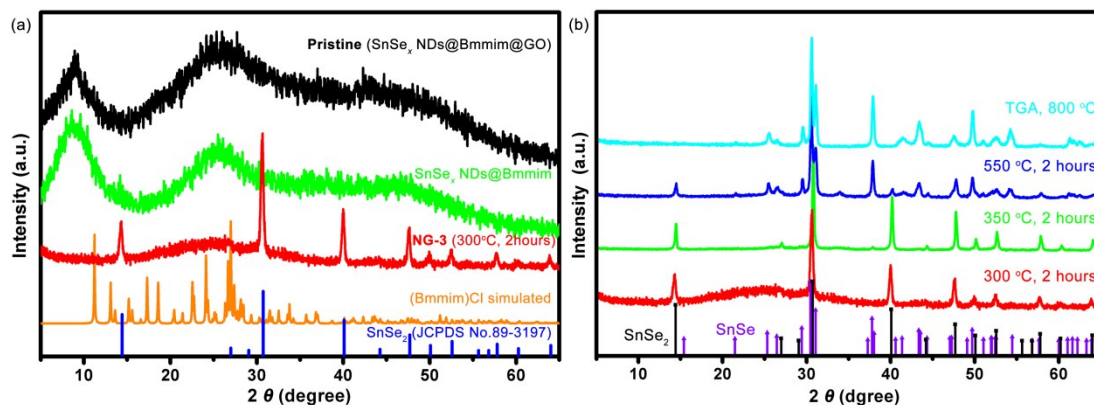


Fig. S4 (a) PXRD patterns of the pristine $\text{SnSe}_x \text{NDs@Bmmim@GO}$ nanocomposite, $\text{SnSe}_x \text{NDs@Bmmim}$, (Bmmim)Cl, product after annealing at 300 °C for 2 hour (NG-3), simulated (Bmmim)Cl and the SnSe_2 phase. (b) PXRD patterns of the products obtained by annealing of NDs@Bmmim@GO nanocomposite at different temperature.

Fig. S5 and S6 show the characterizations (TEM, FTIR, EA) of pristine GO. According to the TEM and HRTEM analyses, the lateral dimension of GO is about 5~6 μm (Fig. S5a). The number of layers of GO is 3~7 layers by counting the edges from the HRTEM images (Fig. S5b).

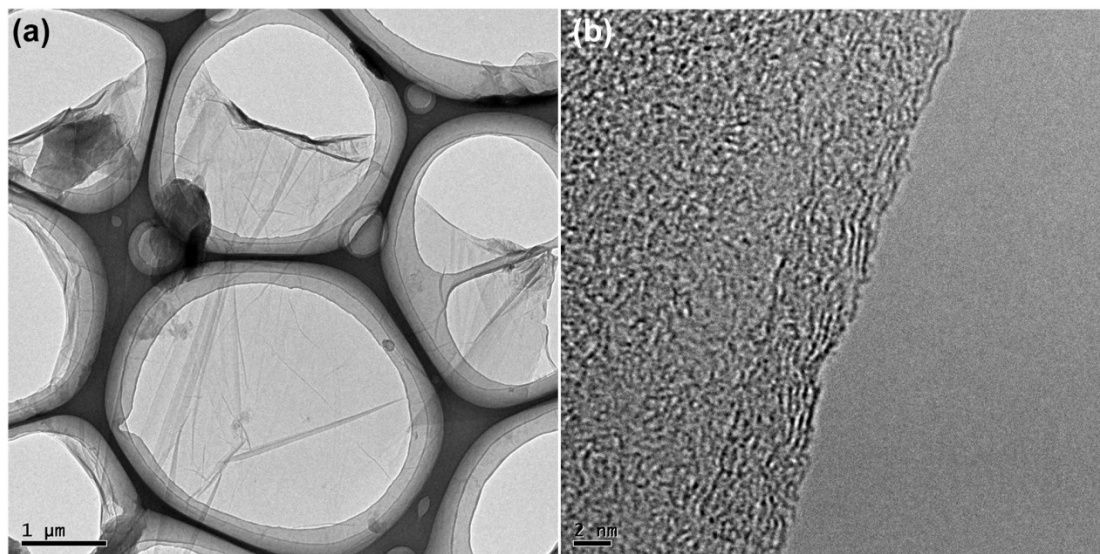


Fig. S5 TEM (a) and HRTEM (b) images of the pristine GO.

The FTIR spectrum of the pristine GO was shown in Fig. S6, which clearly indicates the presence of various functional groups on the sample.^{1, 2}

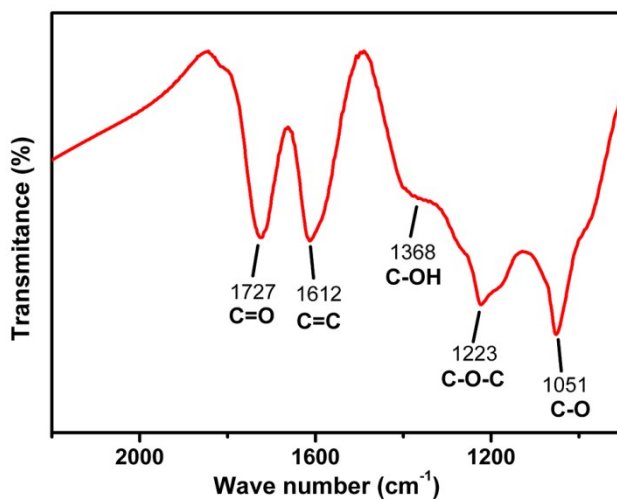


Fig. S6 FTIR spectrum of the pristine GO.

The elemental analysis result of the pristine GO was summarized in Table S1.

Table S1 Elemental analysis result of the pristine GO.

Carbon content (%)	Oxygen content (%)
45.04	50.99

1. J. Liu, C. K. Poh, D. Zhan, L. Lai, S. H. Lim, L. Wang, X. Liu, N. Gopal Sahoo, C. Li, Z. Shen and J. Lin, *Nano Energy*, 2013, **2**, 377-386.
2. X. M. Sun and Y. D. Li, *Angew. Chem., Int. Ed.*, 2004, **43**, 597-601.