Supporting Information for

False Luminescence of Molybdenum Disulfide Quantum Dots from Carbon Dots

Xiaolong Fu,¹ Ju Huang,¹ Xiajun Zhu,¹ Jiefeng Rong,² Zhenyu Lin,¹ Yongqiang Dong*¹ and Fengfu Fu*¹

¹MOE Key Laboratory for Analytical Science of Food Safety and Biology, Fujian Provincial Key Laboratory of Analysis and Detection Technology for Food Safety, College of Chemistry, Fuzhou University, Fuzhou, China. E-mail: dongyq@fzu.edu.cn
²Quanzhou Customs Comprehensive Technology Service Center, Quanzhou 362000, China.

* E-mail: fengfu@fzu.edu.cn; dongyq@fzu.edu.cn

Chemicals. Na₂MoO₄ and L-cys were purchased from Aladdin Industrial Co., Ltd. (Shanghai, China). Hexadecyl trimethyl ammonium bromide (CTAB) and molybdenum sulfide (MoS₂, 99.5% metals basis) were bought from Macklin Biochemical Co., Ltd. (Shanghai, China). All reagents were of analytical grade and without pretreated. The ultrapure water (18.2 MΩ × cm) was used throughout the experiments.

Apparatus. The transmission electron microscope (TEM) and high resolution transmission electron microscopy (HRTEM) images were taken on a Tecnai G2 F20 electronic system (FEI Co., Ltd., USA) at 200 kV. Atomic force microscope (AFM)
patterns were measured by a 5500 Nanoman AFM microscope with tapping mode. X-ray photoelectron spectroscopy (XPS) data were conducted on an ESCALAB 250 XPS system coupled with an Al Kα X-ray source (USA). Ultraviolet-visible (UV-vis) absorption and fluorescence (FL) spectra were recorded on Lambda 750S UV-Vis spectrophotometer (USA) and Hitachi F-4600 spectrofluorophotometer (Kyoto, Japan), respectively. Fourier transform infrared (FTIR) spectra were detected by a Thermo Nicolet 6700 FTIR spectrophotometer (USA). X-ray powder diffraction (XRD) characterizations were carried out on a Rigaku D/max-3 C diffractometer with CuKα radiation (Japan). Raman analysis was obtained using a Renishaw 1000 microspectrometer with excitation wavelength of 632.8 nm (UK).

Synthesis of “MoS$_2$ QDs” and MoS$_2$ nanosheets (MoS$_2$NSs). According to the mostly reported, MoS$_2$ QDs were prepared by “bottom-up” hydrothermal treating the molybdenum and sulfur source. Typically, 0.3 g L-cys and 0.125 g Na$_2$MoO$_4$ were dissolved in 35 mL H$_2$O, and the pH value of the solution was adjusted to 6.5 using 0.1 mol/L HCl. The mixture was ultrasound for 10 min and then transferred into a 100 mL Teflon autoclave, which was heated at 220 ℃ for 24 h. The products were separated by centrifuging at 12000 rpm for 20 min. The suspension labeled as Dots1 (as reported “MoS$_2$ QDs”) and the precipitates were collected for further used. As a control, L-cys (carbon-containing sulfur source) alone was also treated with the identical method above, but in the absence of Na$_2$MoO$_4$. The obtained suspension labeled as Dots2.

The “top-down” exfoliation method by sonication in the presence of H$_2$O or CTAB was also applied to synthesize the monolayer MoS$_2$NSs with different lateral sizes. Briefly, 0.6 g MoS$_2$ powder and 0.65 g CTAB were mixed with 30 mL water, and were ultrasound for 6 h with an ultrasonic power of 350 W and a frequency of 40 KHz. The mixture was centrifuged for 10 min at 3000 rpm to remove the unexfoliated MoS$_2$. The ultrasonic and centrifugal procedures above were repeated two times. Finally, the supernatant was followed by a step-by-step separation using different centrifugation speeds from 5000 to 15000 rpm. The sediments at different
centrifugation speeds were collected and labeled as MoS$_2$NSs5000, MoS$_2$NSs12000, and MoS$_2$NSs15000 corresponding to the centrifugation speeds. The precipitate of hydrothermal Dots1 above was treated by the same “top-down” procedure without CTAB addition. And the product was labeled as MoS$_2$5000, MoS$_2$8000, MoS$_2$12000, and MoS$_2$15000.

Fig. S1 (a) TEM images of Dots2, inset: lattice spacing of Dots2; (b) UV-vis absorption and FL spectra of Dots2, insets: photographs of Dots2 under visible light (left) and 365 nm UV light (right).

Fig. S2 XPS survey spectrum of Dots2.
Fig. S3 C1s high-resolution peak-fitting XPS spectrum of Dots2.

Fig. S4 FTIR spectrum of Dots2.
Fig. S5 XRD spectrum of Dots2.

Fig. S6 XRD behaviors of MoS$_2$5000, MoS$_2$8000, and MoS$_2$12000.
Fig. S7 XPS survey spectrum of MoS$_2$5000.

Fig. S8 C1s high-resolution peak-fitting XPS spectrum of MoS$_2$5000.
Fig. S9 FL measurements of MoS$_2$5000, MoS$_2$8000, MoS$_2$12000, and MoS$_2$15000 (PMT = 850 V).

Fig. S10 The color changes of MoS$_2$5000-12000 (from left to right) without (a) and with (b) H$_2$O$_2$. 
Fig. S11 FL measurements of MoS$_2$5000-15000 with H$_2$O$_2$ (PMT = 850 V).

Fig. S12 (A) FL spectra of the obtained “MoS$_2$ QDs” by electrochemical exfoliation method, insets: photographs of “MoS$_2$ QDs” under visible light (left) and 365 nm UV light (right); (B) FL spectra of products obtained by electrolysis of the ionic liquid alone, insets: photographs of the products under visible light (left) and 365 nm UV light (right).

“MoS$_2$ QDs” were synthesized according to the reported electrochemical exfoliation method.$^3$ For the obtained “MoS$_2$ QDs”, the bright blue light was present under the irradiation of 365 nm UV light (the inset of Fig. S12A). And the emission wavelength red-shifts from 350 to 469 nm with the FL excitation wavelength...
increasing from 280 to 400 nm (Fig. S12A), which is consistent well with the literature. It seems that the “MoS$_2$ QDs” were obtained. For further study the composition of MoS$_2$ QDs, as a control, ionic liquid alone without MoS$_2$ was electrochemical electrolyzed under the same experiment condition. The obtained product also exhibits the excitation-dependent FL emission and blue light (Fig. S12B). It is interesting that whether there is MoS$_2$ precursor or not, the optical properties of the products are quite same, which also fits well with those of CDs.

Reference: