Journal Name

Electronic Supplementary Material (ESI) for Chemical Communications. This journal is © The Royal Society of Chemistry 2022

Supporting Information for

False Luminescence of Molybdenum Disulfide Quantum Dots from Carbon Dots

Xiaolong Fu,^a Ju Huang,^a Xiajun Zhu,^a Jiefeng Rong,^b Zhenyu Lin,^a Yongqiang Dong^{*a} and Fengfu Fu^{*a}

^aMOE 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

^bQuanzhou 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 M $\Omega \times$ 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 K α X-ray source (USA). Ultraviolet-visible (UV-vis) absorption and fluorescence (FL) spectra were recorded on Lamda 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 CuK α radiation (Japan). Raman analysis was obtained using a Renishaw 1000 microspectrometer with excitation wavelength of 632.8 nm (UK).

Synthesis of "MoS₂ QDs" and MoS₂ nanosheets (MoS₂NSs). According to the mostly reported, MoS₂ QDs were prepared by "bottom-up" hydrothermal treating the molybdenum and sulfur source.¹ Typically, 0.3 g L-cys and 0.125 g Na₂MoO₄ were dissolved in 35 mL H₂O, 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 °C for 24 h. The products were separated by centrifuging at 12000 rpm for 20 min. The suspension labeled as Dots1 (as reported "MoS₂ 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 Na₂MoO₄. The obtained suspension labeled as Dots2.

The "top-down" exfoliation method by sonication in the presence of H_2O or CTAB was also applied to synthesize the monolayer MoS_2NSs with different lateral sizes.² 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 $MoS_2NSs5000$, $MoS_2NSs12000$, and $MoS_2NSs15000$ 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_25000 , MoS_28000 , MoS_212000 , and MoS_215000 .



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 MoS₂5000, MoS₂8000, and MoS₂12000.



Fig. S7 XPS survey spectrum of MoS₂5000.



Fig. S8 C1s high-resolution peak-fitting XPS spectrum of MoS₂5000.



Fig. S9 FL measurements of MoS_25000 , MoS_28000 , MoS_212000 , and MoS_215000 (PMT = 850 V).

This journal is $\ensuremath{\mathbb{C}}$ The Royal Society of Chemistry 20xx

Journal Name



Fig. S10 The color changes of $MoS_25000-12000$ (from left to right) without (a) and with (b) H_2O_2 .



Fig. S11 FL measurements of $MoS_25000-15000$ with H_2O_2 (PMT = 850 V).



Fig. S12 UV-vis absorption behaviors of $MoS_2NSs5000$, $MoS_2NSs12000$, and $MoS_2NSs15000$.



Fig. S13 FL measurements of $MoS_2NSs5000$, $MoS_2NSs12000$, and $MoS_2NSs15000$ (PMT = 850 V).



Fig. S14 (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).

"MoS₂ QDs" were synthesized according to the reported electrochemical exfoliation method.³ For the obtained "MoS₂ QDs", the bright blue light was present under the irradiation of 365 nm UV light (the inset of Fig. S14A). And the emission wavelength red-shifts from 350 to 469 nm with the FL excitation wavelength increasing from 280 to 400 nm (Fig. S14A), which is consistent well with the literature. It seems that the "MoS₂ QDs" were obtained. For further study the composition of MoS₂ QDs, as a control, ionic liquid alone without MoS₂ was electrochemical electrolyzed under the same experiment condition. The obtained product also exhibits the excitation-dependent FL emission and blue light (Fig. S14B). It is interesting that whether there is MoS₂ precursor or not, the optical properties of the products are quite same, which also fits well with those of CDs.

Reference:

- 1. Y. Wang and Y. Ni, Anal. Chem., 2014, 86, 7463-7470.
- 2. A. Gupta and S. Vasudevan, J. Phys. Chem. C, 2018, 122, 19243-19250.

Journal Name

3. D. Gopalakrishnan, D. Damien, B. Li, H. Gullappalli, V. K. Pillai, P. M. Ajayan and M. M. Shaijumon, *Chem. Commun.*, 2015, **51**, 6293-6296.