Electronic Supplementary Information for

MOF-Templated Synthesis of Photoluminescent MoS₂ QDs

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1. General Information

Materials used in the experiments were obtained from commercial sources and were used without further purification. The XRD patterns were performed on the D8 advance Diffractometer (Bruker) equipped with CuK α (λ =1.54056 Å) radiation (40 kV, 40 mA). Transmission electron microscopy (TEM) images were collected by 2010-Plus, JEOL. X-ray photoelectron spectroscopy (XPS) measurement was carried out using a ThermoScientific ESCALAB 250 spectrometer with monochromatic Al K α as the excitation source. Raman spectra was collected by Horiba-Jobin-Yvon Raman system with an excitation wavelength at a 532 nm. Atomic force microscope (AFM) images were performed on Asylum Research Cypher AFM. The UV-vis spectra was carried out on a UH4150 (Hitachi) spectrophotometer. Photoluminescence (PL) spectra were recorded on a PE LS-55 spectrometer. Mapping images were collected on Talos F200S.

2. Experimental procedures

The preparation of MOF (MIL-101)

MIL-101 was synthesized according to the previously reported hydrothermal method.¹ Put Chromium (III) nitrate nonahydrate (4.0 g) and terephthalic acid (H₂BDC) (1.66 g) into deionized water (50 mL), stirred the mixture for 30 min. After that, dropped glacial acetic acid (0.58 mL) into the above mixture slowly. Then transferred the mixture to a Teflon-lined stainless steel autoclave with 100 mL volume, heated to 220 °C and kept for 8 h at this temperature. After the reaction completed, the autoclave was allowed to cool down to room temperature, the final green solid product was isolated by centrifugation. Subsequently, the green product was washed with hot DMF (120 °C) and hot ethanol several times. Finally, the product was dried at 150 °C for 12 h under vacuum.

The synthesis of MoS₂ QDs

Accompany the stirring, Molybdenum pentachloride (MoCl₅) of 1184 mg (4 mmol) was dispersed into methanol (10 mL), until MoCl₅ was absolutely dissolved. The following MIL-101 (1000 mg) was added into above mixture. Subsequently, the above mixture was ultrasonicated for 2 h and continuously stirred for 8h. Then, the resulting crude product was centrifuged at 10000 rpm for 10 min, washed with methanol and centrifuged again. The procedure was repeated four times. The final products were dried at 80°C for 12 h.

Well dispersed thiourea (609 mg, 8 mmol) in methanol (30 mL) was then added into the above obtained powder, after the mixture stirred well. Put the mixture into a Teflon-lined stainless steel autoclave with 50 mL volume, heated to 220°C, and maintained for 1 h at this temperature. After the reaction completed, cooling down the autoclave to room temperature naturely, and isolated the solid by centrifugation. Subsequently, respectively used methanol and deionized water to wash out the crude product and dried it at 80°C for 12 h. Finally, MoS₂@MOF powder was obtained. To get the MoS₂ QDs, using the sodium hydroxide solution (500 mL, 60 mmol/L) to etch

the outside MOF, thus releasing the MoS_2 QDs encased in it. After dialysis for 3-4 days to remove the remaining salts, the final MoS_2 QDs was obtained.

3. Supporting figures and tables



Fig. S1 Brunauer-Emmett-Teller (BET) curve of MIL-101, the specific surface area is 2687.656 m^2/g and the average pore diameter is 2.568 nm.



Fig. S2 TEM images of (a) ZIF-8, (b) HKUST-1, (c) MIL-101, (d) ZIF-8 after solvothermal reaction, (e) HKUST-1 after solvothermal reaction, (f) MIL-101 after solvothermal reaction.



Fig. S3 XRD patterns of MIL-101, $MoS_2@MIL-101$ and simulated MIL-101²



Fig. S4 TEM images of the incompletely dissolved $MoS_2@MIL-101$



Fig. S5 HAADF-TEM image of $MoS_2@MIL-101$ for TEM-EDX mapping

Element	Atomic Fraction (%)
S	68.24
Мо	31.76

Table S1 Elemental content of Mo, S from MoS2@MIL-101 according to TEM-EDX.

4. References

1. P. B. Rallapalli, M. C. Raj, S. Senthilkumar, R. S. Somani and H. C. Bajaj, *Environ. Prog. Sustain.* 2016, **35**, 461-468.

2. O. I. Lebedev, F. Millange, C. Serre, G. Van Tendeloo and G. Férey, *Chem. Mater.* 2005, 17, 6525-6527.