Supporting Information

Facile One-Pot Synthesis of Deep Blue Luminescent Lead Bromide Perovskite Microdisks

Zhao Yuan,^a Yu Shu,^a Yu Tian,^b Yan Xin^c and Biwu Ma^{*a,b,d}

^{*a*} Department of Chemical and Biological Engineering, FAMU-FSU College of Engineering, Tallahassee, FL 32310, USA

^b Materials Science Program, Florida State University, Tallahassee, FL 32306, USA

^c National High Magnetic Field Laboratory, Florida State University, Tallahassee FL 32310, USA

^d Department of Chemistry and Biochemistry, Florida State University, Tallahassee, FL, 32306, USA

Table of contents

Experimental Procedures

Figure S1. ¹H NMR spectrum of B-MDs in DMSO.

Figure S2. ¹H NMR spectrum of T-MDs in DMSO.

Figure S3. TGA heating curves of B-MDs and its corresponding 1st derivative.

Figure S4. TGA heating curves of T-MDs and its corresponding 1st derivative.

Table S1. Composition of the component molar ratio in B-MDs.

- Table S2. Composition of the component molar ratio in T-MDs.
- Figure S5. TEM images of B-MDs and the corresponding electron diffraction patterns.
- Figure S6. TEM images of T-MDs and the corresponding electron diffraction patterns.
- Figure S7. 3D AFM images of B-MDs.
- Figure S8. 3D AFM images of T-MDs.
- Figure S9. Photoluminescent spectra of thin films based on 2D perovskite bulk samples.

Experimental Procedures

Materials. Lead(II) bromide (99.999%), octylamine (99%), benzylamine (99%), 2thiophenemethylamine and hydrobromic acid (48%) were purchased from Sigma-Aldrich. Acetone (99.5%) was purchased from VWR. *N*,*N*-Dimethylformamide (99.9%), toluene (99.9%) and hexane (98.5%, mixture of isomers) were purchased from Sigma-Aldrich. All reagents and solvents were used without further purification unless otherwise stated. Spectroscopic grade solvents were used in the UV-Vis and photoluminescence spectroscopic measurements.

Synthesis of 2D organometal halide perovskite microdisks. 0.1 mmol lead(II) bromide (0.10 mmol, 36.7 mg) was dissolved in 200 μ L DMF with 26.4 μ L octylamine (0.16 mmol) and 26.2 μ L benzylamine (0.24 mmol) or 24.7 μ L 2-thiophenemethylamine (0.24 mmol) followed by adding 50.0 μ L hydrobromic acid aqueous solution (48 wt.%, 0.44 mmol) to form a clear precursor solution, which was subsequently dropped into 2 mL hexanes with vigorously stirring for 5 min at room temperature. The white 2D perovskite microdisks were induced right after the addition of 3 mL acetone followed by centrifugation to remove the unreactive materials in the clear supernatant, affording a white powder in a 84% yield (63 mg for B-MDs) and a 81% yield (61 mg for T-MDs) after dried under vacuum.

Synthesis of 2D organometal halide perovskite bulk sample. The corresponding bulk perovskite samples were prepared by following the same procedure for perovskite microdisks without using the octylamine ligand.

Nuclear magnetic resonance (¹H-NMR). ¹H NMR spectra were acquired at room temperature on Bruker AVANCE III NMR Spectrometers with a 500 MHz Bruker magnet. All chemical shifts (δ) were reported in ppm relative to tetramethylsilane (TMS).

Thermogravimetry analysis (TGA). TGA was carried out using a TA instruments Q50 TGA system. The samples were heated from 22 to 800 °C with at a rate of 5 °C/min, under a nitrogen flux of 100 mL/min.

X-ray diffraction (XRD). The XRD analysis was performed on Panalytical X'PERT Pro Powder X-Ray Diffractometer using Copper X-ray tube (standard) radiation at a voltage of 40 kV and 40 mA, and X'Celerator RTMS detector. The diffraction pattern was scanned over the angular range of 3-70 degree (20) with a step size of 0.02, at room temperature. **Transmission Electron Microscopy images (TEM).** Microstructural characterization was performed using transmission electron microscopy (TEM), on a JEOL JEM-ARM200cF at 200kV. Low intensity illumination and fast acquisition time were used during data collection to avoid beam damage. TEM samples were prepared by depositing a few drops of the perovskite solution on a carbon film supported copper grid (200 mesh); the samples were subsequently dried overnight.

Atomic Force Microscopy images (AFM). AFM measurements were conducted using Bruker Icon. All measurements were performed in the standard tapping mode with OTESPA-R3 tips from Bruker.

UV-visible measurements. UV-Vis spectra were conducted at room temperature using a quartz spectrometer cuvette on a Varian Cary 100 Bio UV-Visible spectrophotometer.

Photoluminescence steady state studies. Steady-state photoluminescence spectra were obtained at room temperature on a Varian Cary Eclipse Fluorescence spectrophotometer. All the data were acquired using a 1-cm semi-micro quartz cuvette. The emission spectra of the perovskites dispersed in toluene were measured under air atmosphere (unless otherwise indicated).

Time resolved photoluminescence. Time resolved photoluminescence data were collected at room temperature using a Horiba JY Fluoromax-4 Fluorometer. The dynamics of emission decays was monitored by using the time-correlated single photon counting (TCSPC) mode (1024 channels; 50 ns window) with data collection for 10,000 counts. Excitation was provided by a 370-nm LED with a repetition rate of 1 MHz. The emission decay data were then analyzed using the DAS6 software. The average lifetime was obtained from the tri-exponential decays according to the equation 3.

 $\tau_{ave} = \sum \alpha_i \tau_i^2 / \sum \alpha_i \tau_i, i = 1, 2, 3$ (3)

where τ_i represents the decay time and α_i represents the amplitude of each component.

Photoluminescence quantum yields (PLQYs). For photoluminescence quantum yield measurement, the samples were excited at 360 nm. Absorbance at the excitation wavelength was kept below 0.1 to minimize the inner-filter effect. The fluorescence quantum yields were determined by comparing the integrated area of the corrected emission spectrum with that of the reference – quinine bisulfate ($\phi = 0.54$ in 1 N H₂SO₄).¹



Figure S1. ¹H NMR spectrum of B-MDs in DMSO.



Figure S2. ¹H NMR spectrum of T-MDs in DMSO.



Figure S3. (a) TGA heating curves of B-MDs. (b) The corresponding 1st derivative.



Figure S4. (a) TGA heating curves of T-MDs. (b) The corresponding 1st derivative.

Table S1. Composition of the component molar ratio in B-MDs according to TGA and ¹H NMR data.

Reagents	Starting material (mmol)	Component molar ratio (based on PbBr ₂)
OABr	0.16	0.07
BABr	0.24	1.95
PbBr ₂	0.10	1.00

Table S2. Composition of the component molar ratio in T-MDs according to TGA and ¹H NMR data.

Reagents	Starting material (mmol)	Component molar ratio (based on PbBr ₂)
OABr	0.16	0.10
BABr	0.24	2.02
PbBr ₂	0.10	1.00



Figure S5. TEM images of B-MDs (top) and the corresponding electron diffraction patterns (bottom).



Figure S6. TEM images of T-MDs (top) and the corresponding electron diffraction patterns (bottom).



Figure S7. 3D AFM images of individual (a) and aggregations (b) of B-MDs.



Figure S8. 3D AFM images of individual (a) and aggregations (b) of T-MDs.



Figure S9. Photoluminescent spectra of thin films based on 2D perovskite bulk samples.



Figure S10. Powder XRD patterns of B-MD (a) and T-MD (b) powder samples.



Figure S11. Photoluminescence spectra of (a) B-MDs and (c) T-MDs during 30-minute irradiation from a hand-held UV lamp (365 nm, 4 W) under argon atmosphere; Photoluminescence intensity at maximum of (b) B-MDs and (d) T-MDs dispersed in toluene as a function of the irradiation time.

1. S. Fery-Forgues and D. Lavabre, J. Chem. Ed., 1999, **76**, 1260-1264.