

# Supporting Information

## Efficient Preparation of Small-Sized Transition Metal Dichalcogenide Nanosheets by Polymer-Assisted Ball Milling

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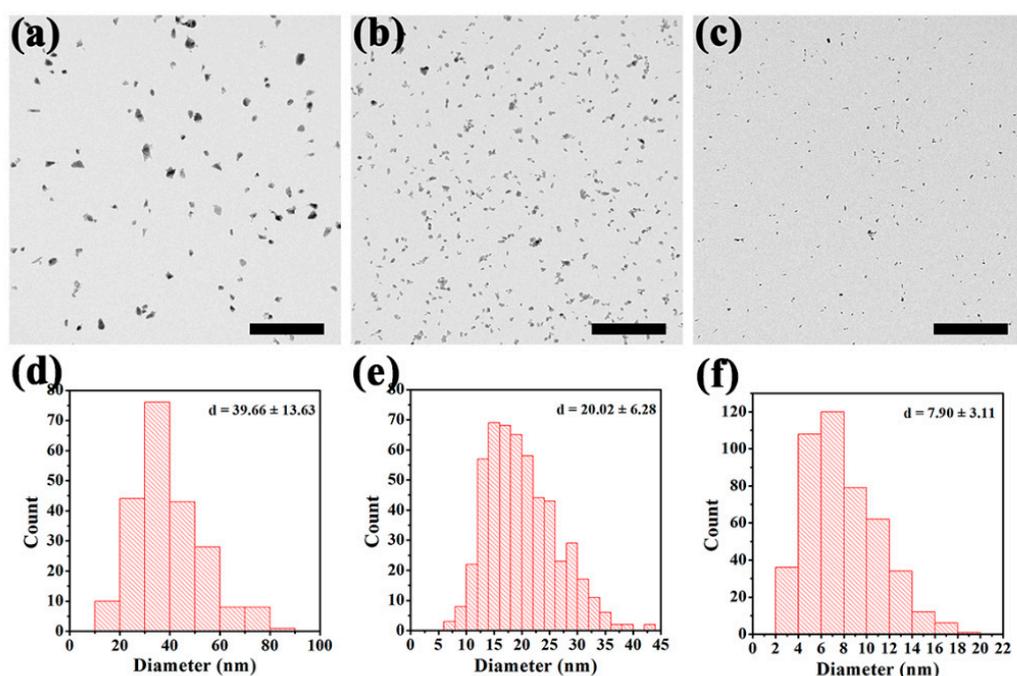


Figure S1 Large-scale TEM images of WSe<sub>2</sub> NSs prepared by CMC-assisted ball-milling at 650 rpm for 12 h and centrifuged at different conditions: (a) 10000 rpm for 1 h, WSe<sub>2</sub>-Low; (b) 16000 rpm for 1.5 h, WSe<sub>2</sub>-Medium; (c) 21000 rpm for 4 h, WSe<sub>2</sub>-High. Scale bar: 500 nm. Size statistics of WSe<sub>2</sub>-Low (d), WSe<sub>2</sub>-Medium (e), and WSe<sub>2</sub>-High (f) based on more than 100 nanosheets.

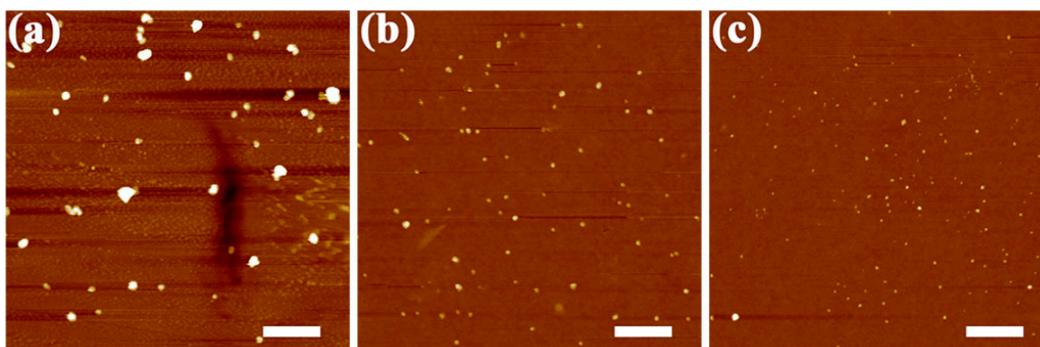


Figure S2 Large-scale AFM images of WSe<sub>2</sub> NSs prepared by CMC-assisted ball-milling at 650 rpm for 12 h and centrifuged at different conditions: (a) WSe<sub>2</sub>-Low; (b) WSe<sub>2</sub>-Medium; (c) WSe<sub>2</sub>-High. Scale bar: 500 nm.

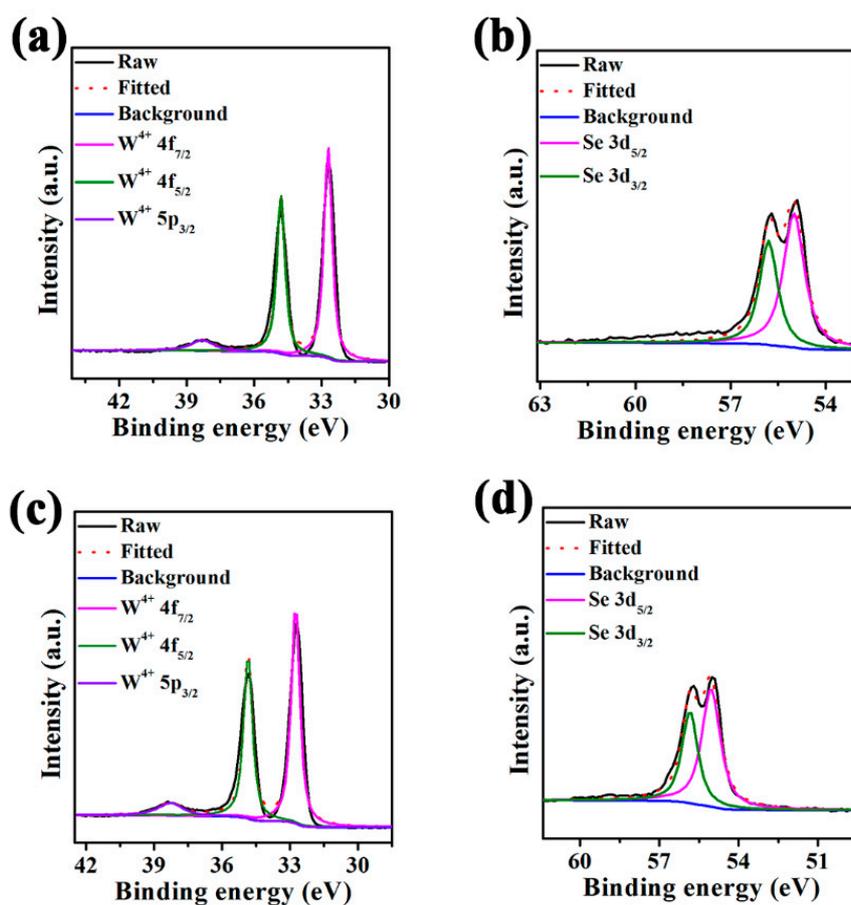


Figure S3 XPS spectra of WSe<sub>2</sub> NSs prepared by CMC-assisted ball-milling at 650 rpm for 12 h: (a–b) WSe<sub>2</sub>-Medium, (c–d) WSe<sub>2</sub>-High.

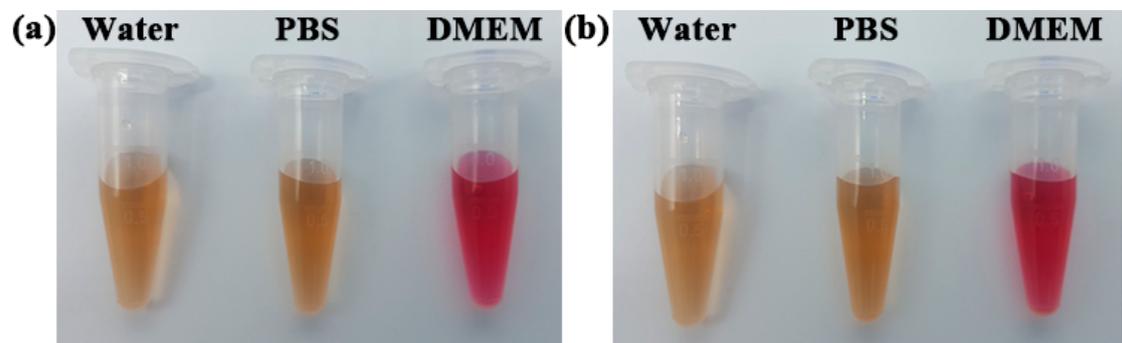


Figure S4 Photographs of CMC-WSe<sub>2</sub> NSs dispersed in water, PBS, and DMEM for different times: (a) 0 h and (b) 24 h.

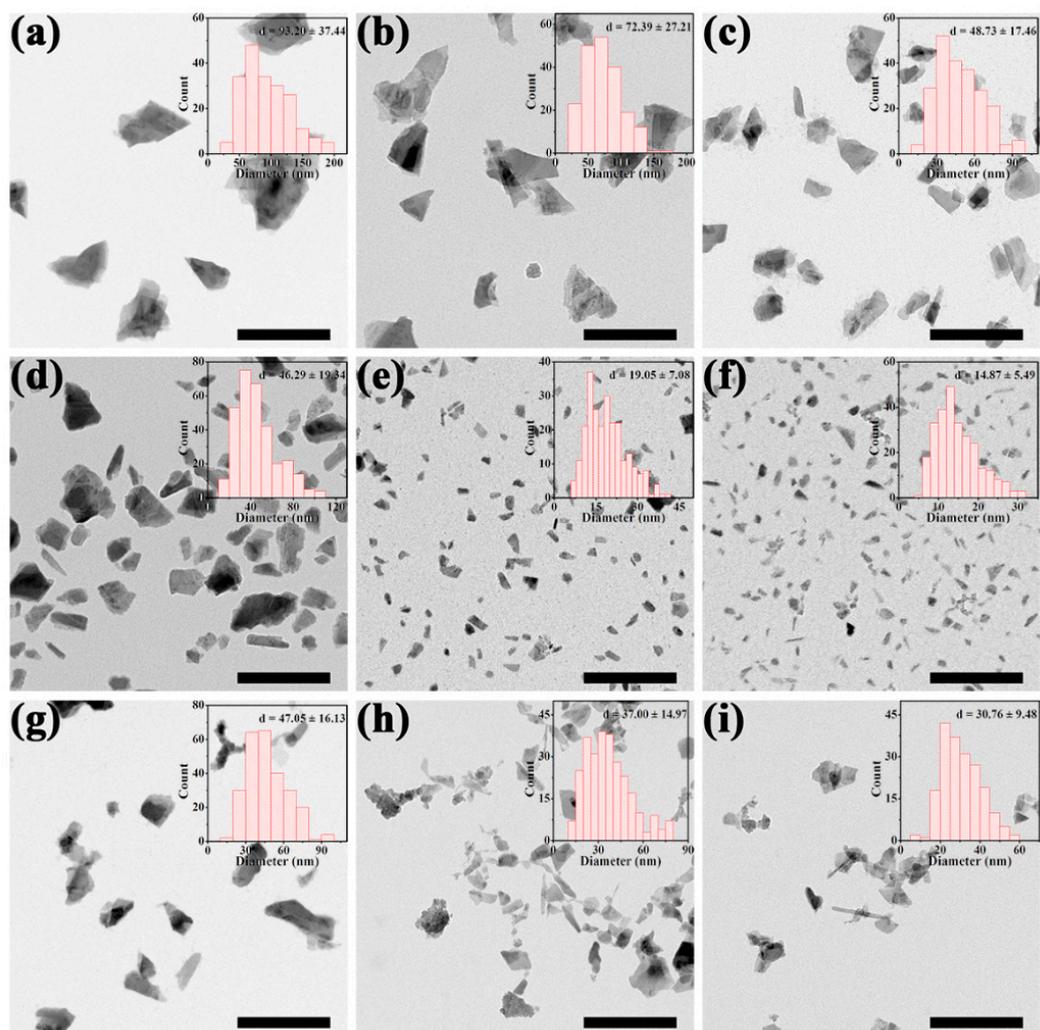


Figure S5 TEM images of WSe<sub>2</sub> NSs prepared by ball-milling with different polymers

at different centrifugal conditions: (a) F127, 5000 rpm for 20 min, WSe<sub>2</sub>-Low; (b) F127, 7500 rpm for 20 min, WSe<sub>2</sub>-Medium; (c) F127, 10000 rpm for 30 min, WSe<sub>2</sub>-High; (d) PVP, 10000 rpm for 1 h, WSe<sub>2</sub>-Low; (e) PVP, 16000 rpm for 2 h, WSe<sub>2</sub>-Medium; (f) PVP, 21000 rpm for 4 h, WSe<sub>2</sub>-High; (g) PEG, 5000 rpm for 20 min, WSe<sub>2</sub>-Low; (h) PEG, 7500 rpm for 20 min, WSe<sub>2</sub>-Medium; (i) PEG, 10000 rpm for 30 min, WSe<sub>2</sub>-High. Scale bar: 200 nm. The insets show the size statistics of WSe<sub>2</sub> NSs based on more than 100 nanosheets from TEM images.

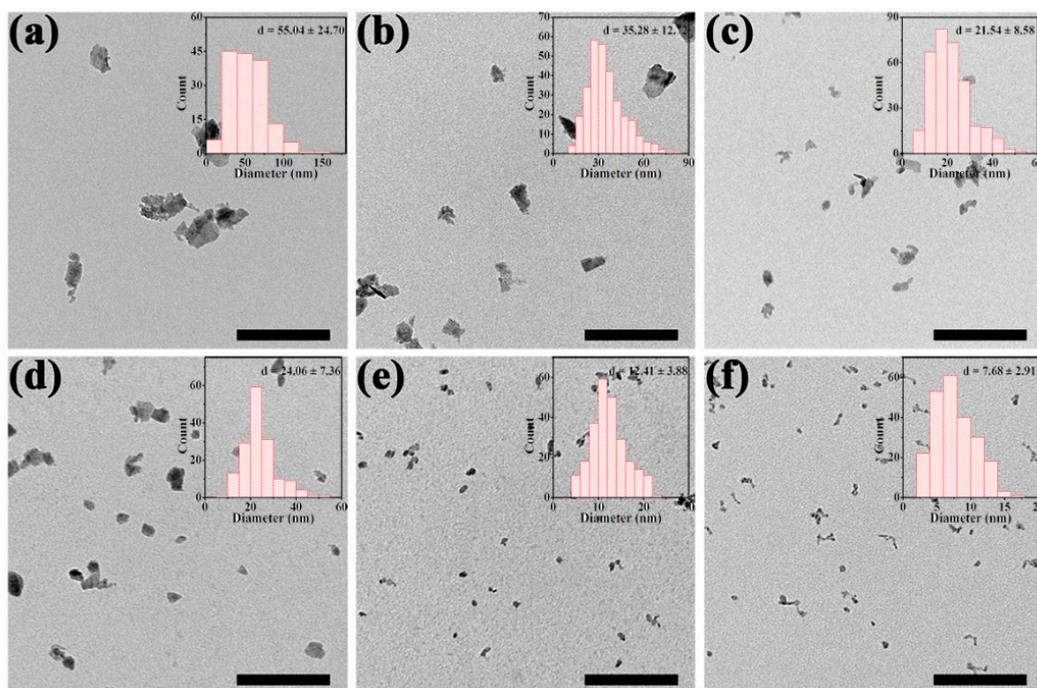


Figure S6 TEM images of WSe<sub>2</sub> NSs prepared by CMC-assisted ball-milling for different times after gradient centrifugation: (a) 6 h, WSe<sub>2</sub>-Low; (b) 6 h, WSe<sub>2</sub>-Medium; (c) 6 h, WSe<sub>2</sub>-High; (d) 24 h, WSe<sub>2</sub>-Low; (e) 24 h, WSe<sub>2</sub>-Medium; (f) 24 h, WSe<sub>2</sub>-High. Scale bar: 200 nm. The insets are size statistics of WSe<sub>2</sub> NSs based on more than 100 nanosheets from TEM images.

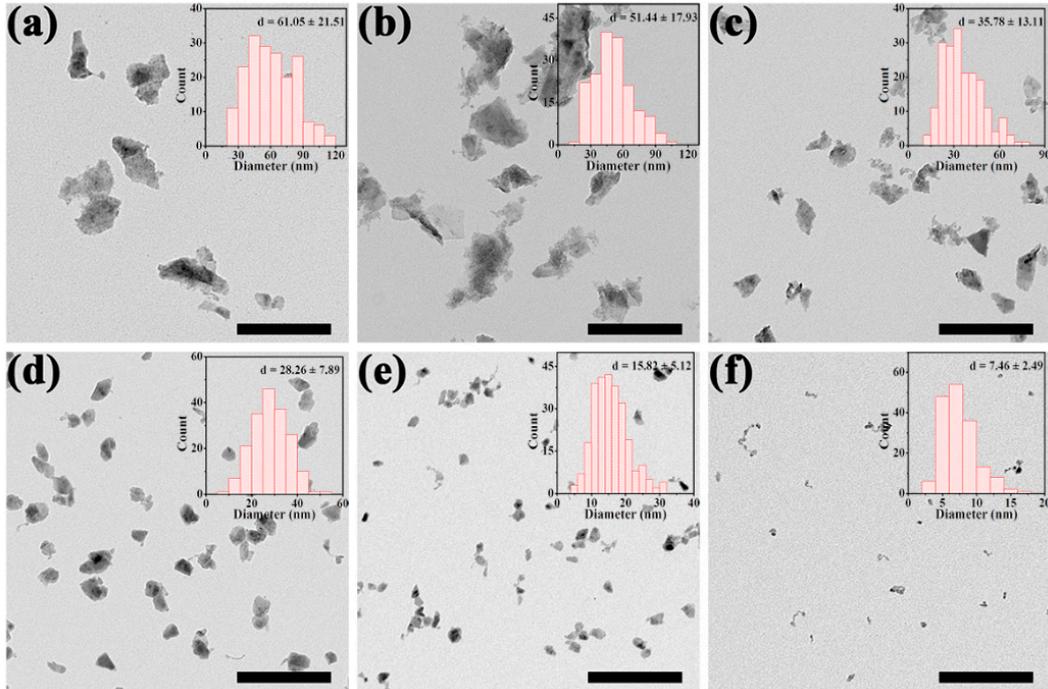


Figure S7 TEM images of WSe<sub>2</sub> NSs prepared by CMC-assisted ball-milling with different rotation speeds after gradient centrifugation: (a) 400 rpm, WSe<sub>2</sub>-Low; (b) 400 rpm, WSe<sub>2</sub>-Medium; (c) 400 rpm, WSe<sub>2</sub>-High; (d) 800 rpm, WSe<sub>2</sub>-Low; (e) 800 rpm, WSe<sub>2</sub>-Medium; (f) 800 rpm, WSe<sub>2</sub>-High. Scale bar: 200 nm. The inset of size statistics is calculated based on more than 100 nanosheets from TEM images.

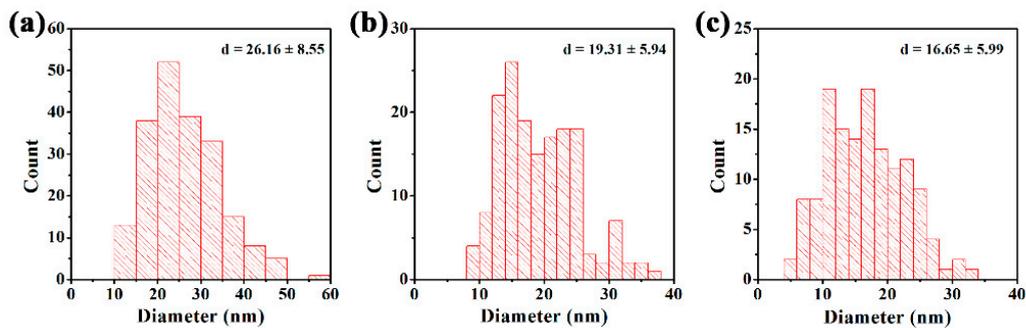


Figure S8 Size statistics of (a) MoS<sub>2</sub>, (b) MoSe<sub>2</sub>, and (c) WS<sub>2</sub> NSs prepared by CMC-assisted ball milling.

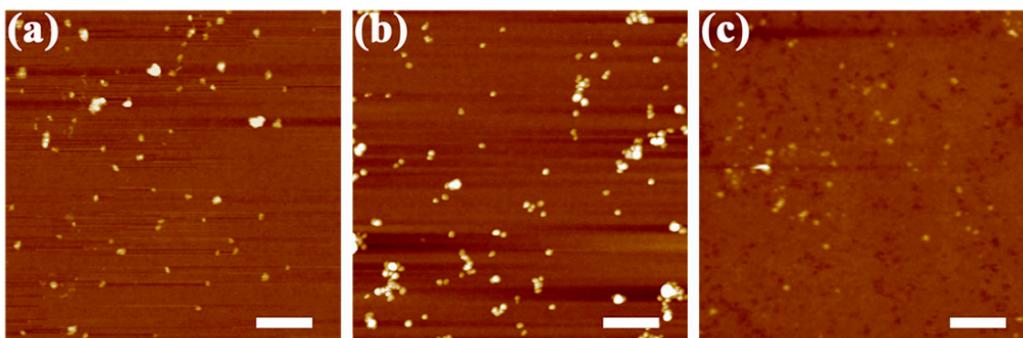


Figure S9 Large-scale AFM images of (a) MoS<sub>2</sub>, (b) MoSe<sub>2</sub>, and (c) WS<sub>2</sub> NSs prepared by CMC-assisted ball-milling. Scale bar: 500 nm.

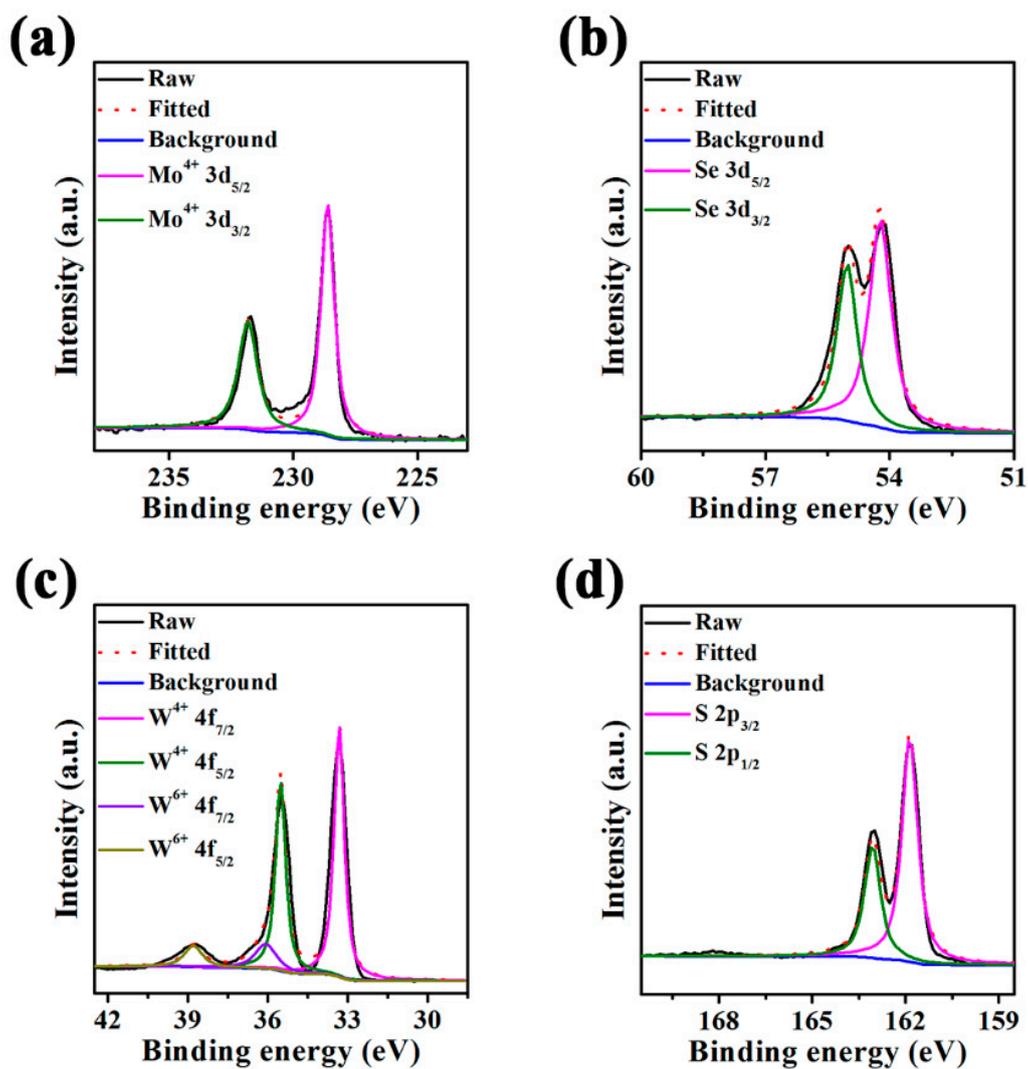


Figure S10 High-resolution XPS spectra of Mo 3d (a) and Se 3d (b) core level energy regions for MoSe<sub>2</sub>, and W 4f (c) and S 2p (d) core level energy region for WS<sub>2</sub>.

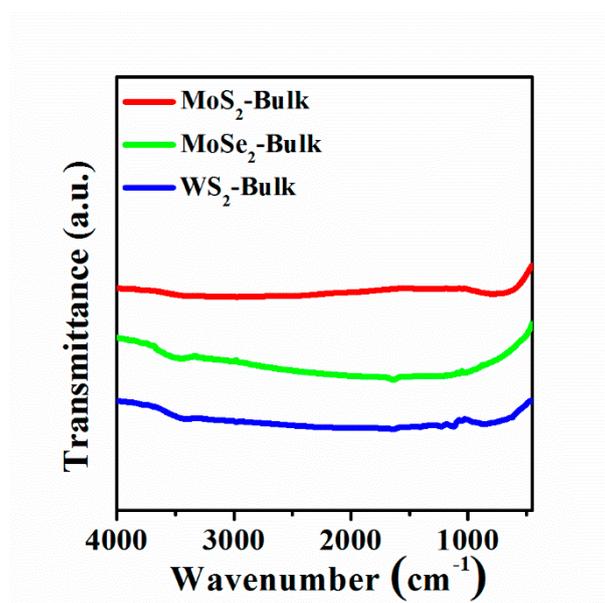


Figure S11 FT-IR spectra of bulk MoS<sub>2</sub>, MoSe<sub>2</sub>, and WS<sub>2</sub>.