Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information For

Simultaneous Exfoliation and Colloidal Formation of Few-Layer Semiconducting MoS_2 sheets in Water

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Experimental Section

Material preparation. Molybdenum (Ⅳ) sulfide (MoS_2) powder, tungsten sulfide (WS_2) powder and Molybdenum Selenide ($MoSe_2$) powder from alfa, 1-pyrenebutyric acid and sodium hydroxide from Innochem and tetrabutylammonium hydroxide solution from Sigma-aldrich were used as-received. The preparation process started by mixing the 1-pyrenebutyric acid and sodium hydroxide with 30 mL distilled water to produce 1-pyrenebutyric acid sodium salt (PyB-Na) in 500 mL glass beaker. When we used 1 mmol/mL tetrabutylammonium hydroxide instead, we obtain 1-pyrenebutyric acid tetrabutylammonium salt (PyB-TBA). Then MoS_2 powder was added into the glass beaker and kept still for 24 h at room temperature for the purpose of intercalation of the PyB molecules. Magnetic stirring for 48 h instead of keeping still for 24 h was used as an optimization. Subsequently, 270 mL distilled water was added into beaker for shear mixing which was performed using a L5M high-shear laboratory mixer from Silverson Machines, in ice-bath. After the mixer was run at 6000 rpm for a given period, the dispersions were kept still for 4 h and the top 200 mL suspension was decanted. The decanted dispersions were centrifuged for 10 min at different centrifugal force, where approximately the top 50% of the supernatant was collected by pipet for spectroscopic characterizations.

Characterizations. UV-vis spectra of the exfoliated samples were measured on Agilent Technologies Cary100 Series UV-Vis Spectrophotometer. Scanning electron microscope (SEM) images were collected on a Hitachi SU8010 at 3 kV. High resolution transmission electron microscope (HRTEM) images were collected on a FEI Talos F200X at 200 kV. For the HRTEM measurement a drop of the MoS_2 dispersion was casted on a holey carbon grid (400 meshes). Atom force microscope (AFM) images were collected in tapping mode to characterize the sheet thickness and size distributions. For AFM measurement, the decanted dispersion was further filtered using a 0.45 μm Nylon (polyamide) membrane to remove extra PyB salt and the flakes remained on the membrane were re-dispersed in water, which was subsequently cast on oxidized silicon and drying on hotplate. Spray-coating was used to deposit the exfoliated MoS_2 sheets onto the surfaces of a Si/SiO_2 substrate at 105°C. The samples were soaking in DI-water for 15 min after spray-coating and then drying at 120 ℃ on hot plate. Raman spectroscopy was conducted on a Renishaw Raman spectroscope with laser wavelength of 532 nm and 20× lens. The concentration of exfoliated MoS2 sheets in the dispersions was determined by drying and weighing. X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) were employed to characterize the phase of the exfoliated MoS_2 and the role of the PyB salt in the exfoliation.

Figure S1. MoS_2 dispersions in water with (left) and without PyB-Na (right) right after shear mixing.

S1 Optimization of exfoliation condition

The influences of the dosage of initial MoS_2 powder and the time of shear mixing on the concentration of final MoS_2 dispersion were investigated in detail. Figure S2a and S2b illustrates the influences of initial MoS_2 dosage on the concentration of the final MoS_2 dispersions. The figure in Figure S2a for each line represents the amount of MoS_2 used. In Figure S2b, the highest concentration of MoS_2 dispersion could be obtained when the amount of the initial dosage of MoS_2 powder were 4.5 g. Similarly, Figure S2c and S2d illustrates the influences of the time of shear mixing. The figure in Figure S2c for each line represents the time of shear. In Figure S2d, the highest concentration of MoS_2 dispersion could be obtained when the time of shear mixing of MoS_2 powder were 3.0 h. Considering the quality of the MoS_2 nanosheets, the MoS_2 nanosheets obtained by shearing 1.0 h were characterized.

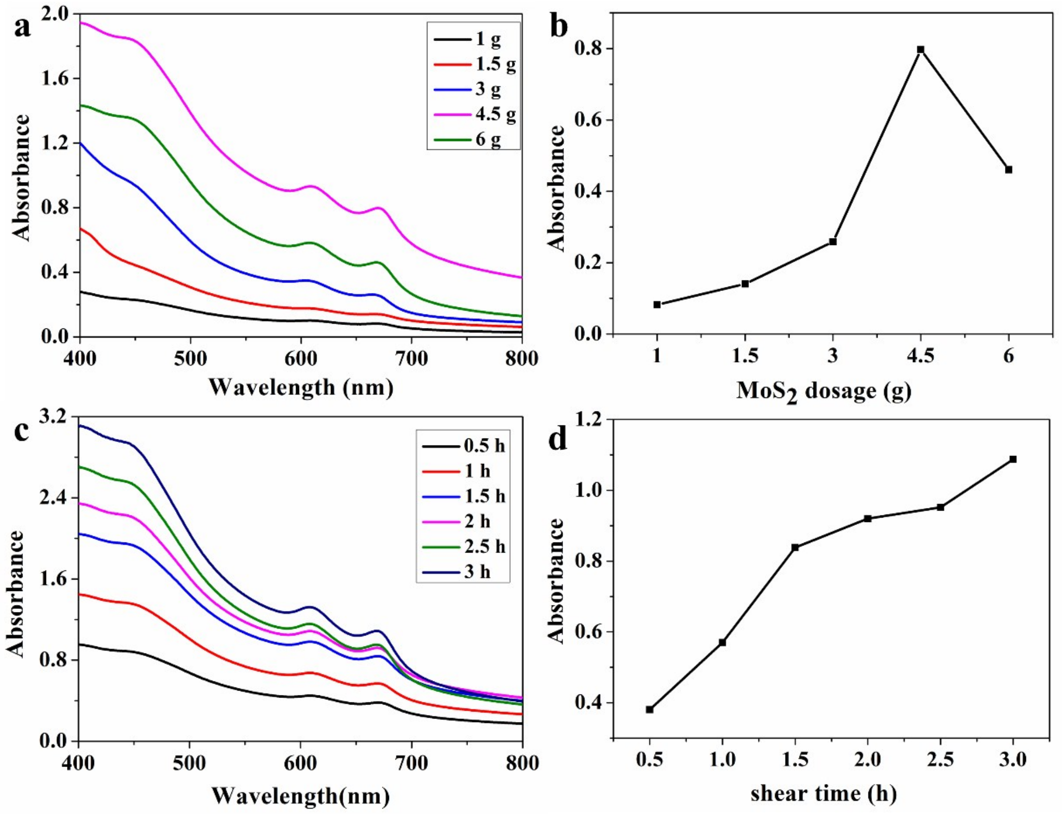

Figure S2. The UV-vis spectra of MoS_2 dispersions in water assisted with different initial dosage of MoS_2 powder and with the different shear time.

S2 Stability test

Detecting the dependence of concentration on time is a useful method for examine the stability of the MoS_2 nanosheets dispersions. We measured the absorbance of the dispersions for 32 days with the interval of eight days. Figure S3 shows the concentration retention versus time of the MoS_2 dispersions with PyB-Na. The concentration retained 89% after 32 days. It indicates that these MoS_2 dispersions are stable without significant aggregation at least for one month.

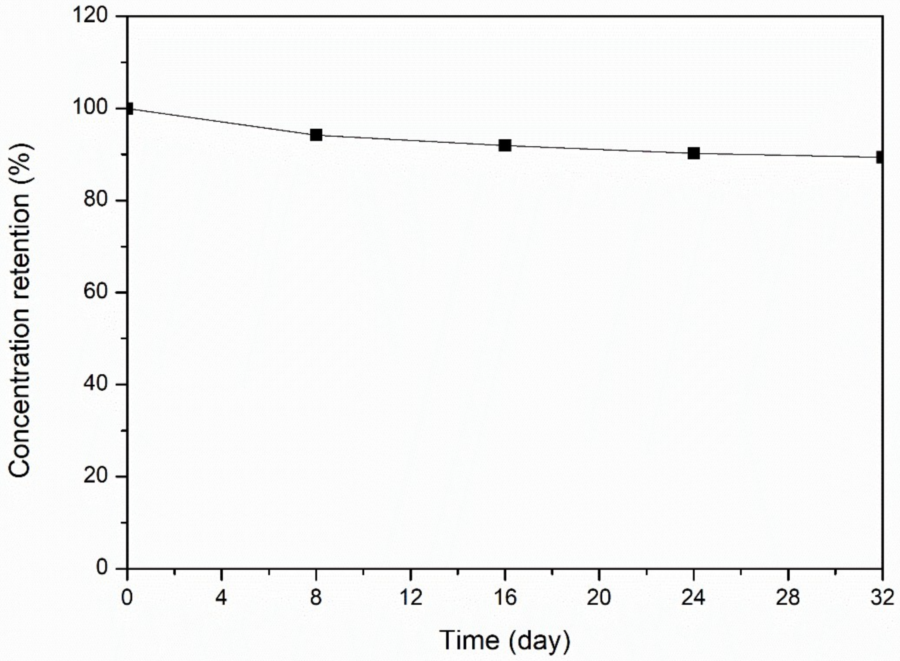

Figure S3. Absorbance retention of MoS_2 dispersions as a function of time

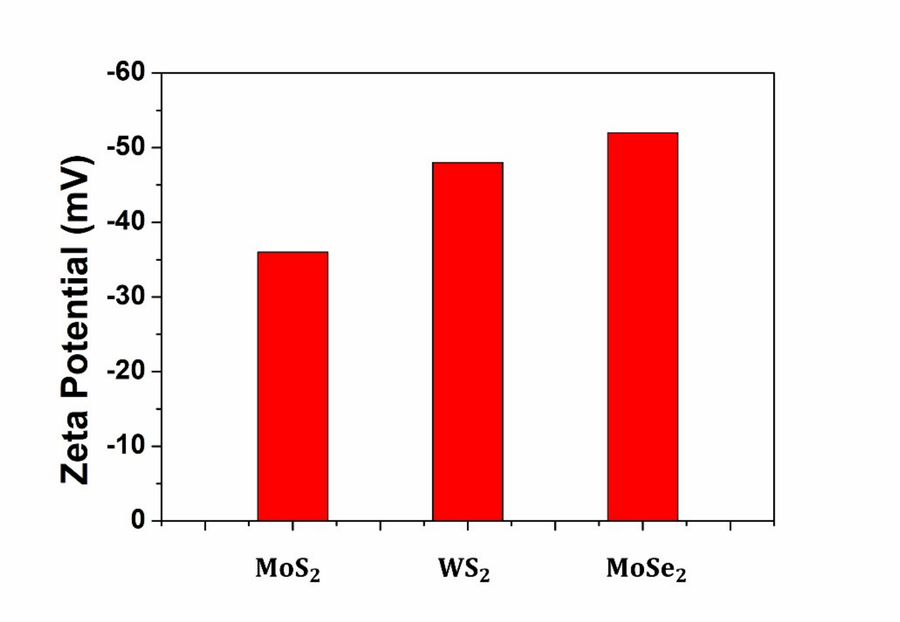

Figure S4. Zeta potential of exfoliated MoS_2, WS_2 and $MoSe_2$ sheets in water

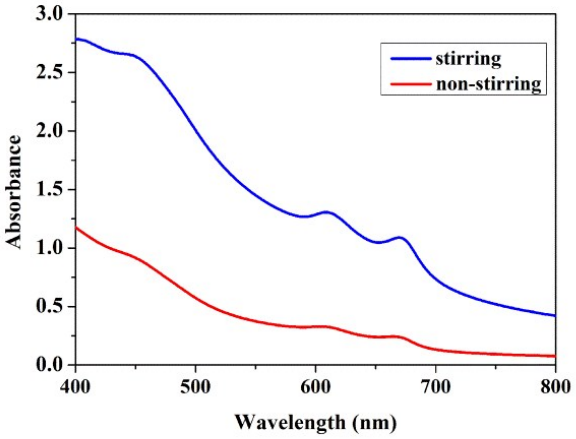

Figure S5. The UV-vis spectra of MoS_2 dispersions in water by previous magnetic stirring (blue), and by previous still standing (red).

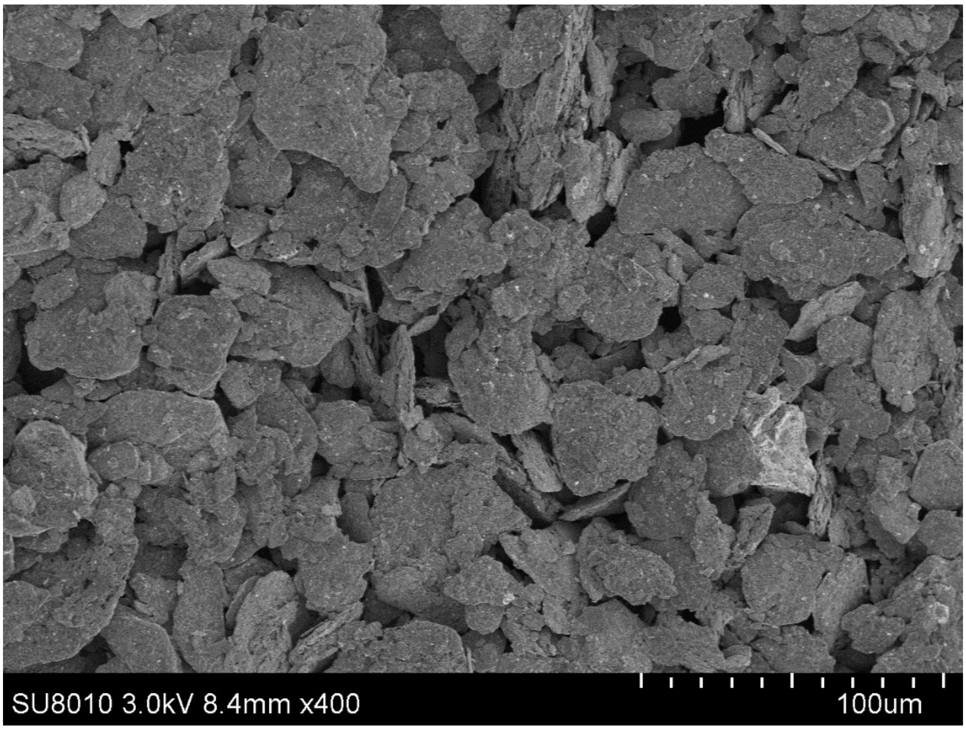

Figure S6. SEM image of bulk MoS_2

Figure S7. Optical photograph of exfoliated WS_2 (left) and $MoSe_2$ (right)

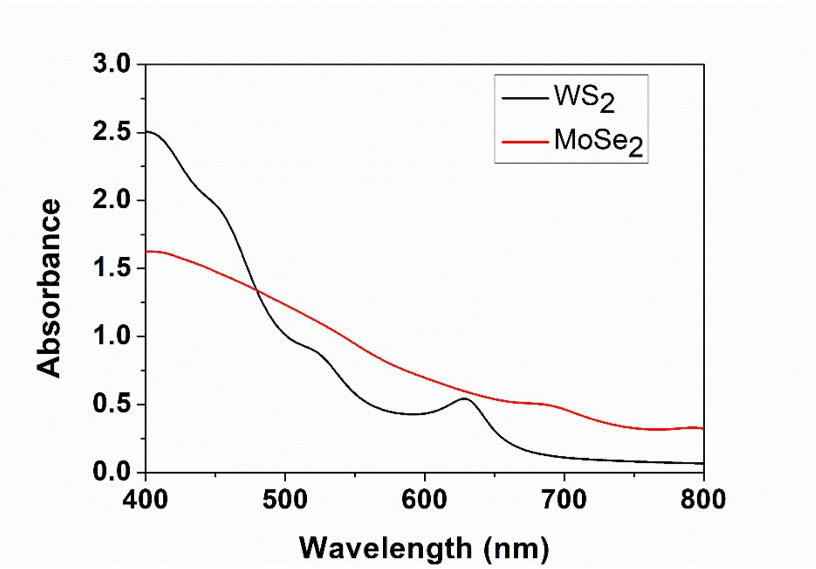

Figure S8. The UV-vis spectra of exfoliated WS_2 and $MoSe_2$