Electronic Supplementary Material (ESI) for Nanoscale Advances.

Supporting Information

High Conversion Continuous Flow Exfoliation of 2D MoS_2

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Experimental section:

Materials:

MoS_2 powder was purchased from Aladdin (particle sizes ~1.5 mm). Dimethylformamide (DMF) (99%) and N-methyl-2-Pyrrolidone (NMP) were purchased from Sigma Aldrich, and ethanol (99%) was purchased from Alfa Aesar. Ultrapure water (18.2 MΩ cm, Milli-Q Direct 8) was used in all studies.

Synthesis of exfoliated MoS_2 sheets:

Exfoliating of bulk MoS_2: As received MoS_2 (100 mg) with lateral dimension size of ~1.5 mm was dispersed in a 1:1 mixture of ethanol and water. The solution was then sonicated for 30 mins (ultrasonic processor KH5200E) whereupon it was injected into the VFD tube which was rotating at 8k rpm, at a flow rate 0.45 mL/min. Thereafter the product was drop casted onto clean silicon wafer substrates for characterisation, which included SEM (scanning electron microscopy) and AFM (atomic force microscopy) studies, and was found to be comprised primarily of 2D MoS_2 nanosheets. The isolated yield for the exfoliated material formed under continuous flow was 75 % after centrifuge the collected product at 9980 g for 15 min . This was based on the concentration and volume of the as-received MoS_2 feed (5 $mg.mL^{-1}$) relative to the quantity of isolated MoS_2 after removing the solvent(s) *in vacuo*, for a particular volume of liquid processed through the VFD under steady state flow conditions.

Characterization:

The exfoliated MoS_2 sheets were characterized using Raman spectroscopy (Witec Raman microscope spectrometer, 532 nm). The morphology and chemical composition were further characterized using SEM for a FEI Quanta 450 High Resolution Field Emission instrument operating with a voltage of 10 kV. AFM studies used a Nanoscope 8.10 instrument operating under tapping mode, and TEM (transmission electron microscopy) was conducted on a TECNAI 20 microscope operating at 120 and 200 kV. STEM imaging and compositional mapping were conducted using an aberration-corrected FEI Titan Themis TEM operating at 200 kV equipped with an energy dispersive X-ray spectroscopy (EDX) detector. X-ray powder diffraction (XRD) data were collected using a Bruker Advanced D8 diffractometer (capillary stage) using Co-Kα radiation (λ = 1.7889 Å, 35 kW/28 mA, 2θ = 10–90°). X-ray photoelectron spectroscopy (XPS) data was acquired using an UHV instrument with a Phoibos 100 hemispherical analyser (SPECS) operating under a base pressure of 10^{-9} mbar with the incident

radiation as a non-monochromatic X-ray source with a Mg anode (12 kV–200 W, Kα line with an excitation energy of 1253.6 eV).

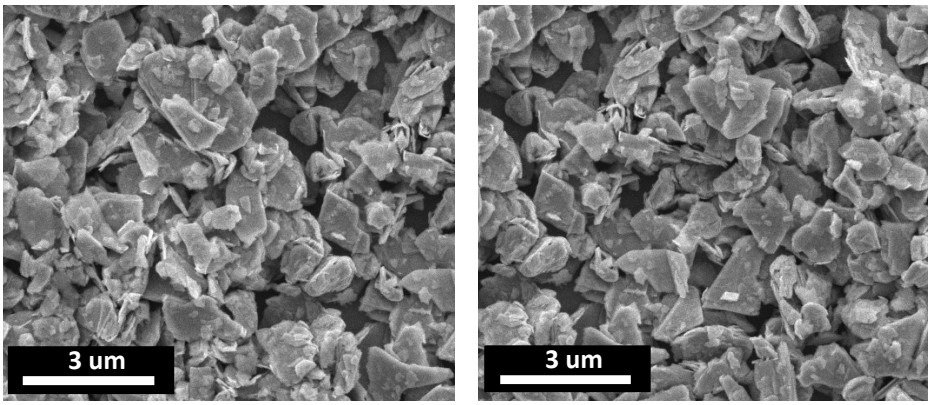

Figure S1. SEM images of MoS_2 before processing in the VFD.

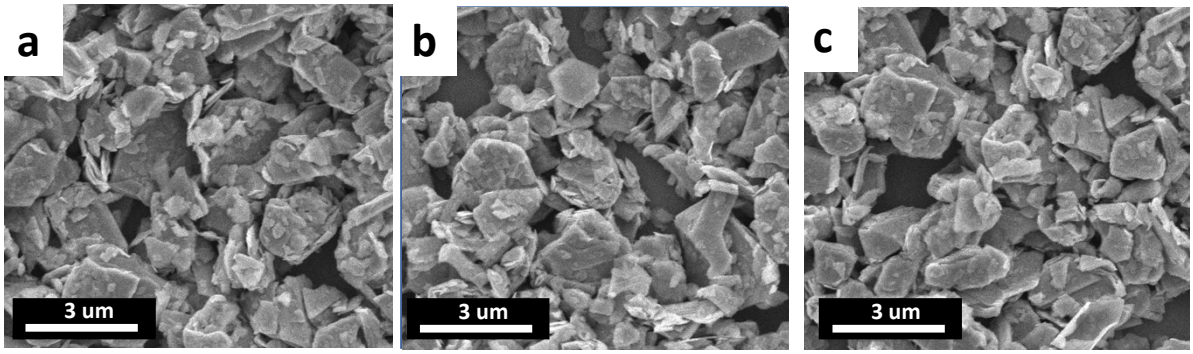

Figure S2. SEM images of MoS_2 processed in the VFD at 8k rpm (optimised rotational speed), under continuous flow at a flow rate 0.45 mL.min^{-1}, tilt angle θ 45º, with the concentration of the as received MoS_2 at 5 mg. mL^{-1} (optimised concentration) dispersed (a) DMF, (b) NMP and (c). Ethanol/Water/DMF

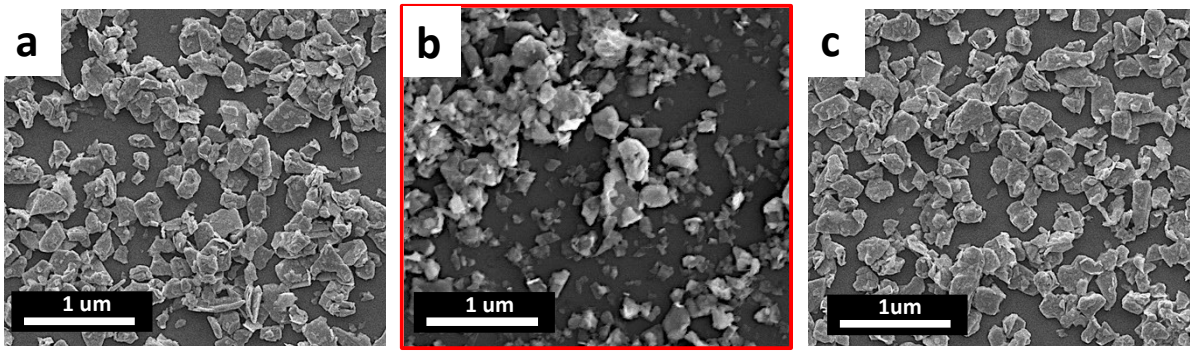

Figure S3. SEM images of MoS_2 processed in the VFD at 8k rpm rotational speed, under continuous flow at a flow rate 0.45 mL/min, tilt angle θ 45º, in different concentration at (a) 2 mg. mL^{-1}, (b) 5 mg. mL^{-1} (optimised concentration) and (c) 10 mg. mL^{-1}.

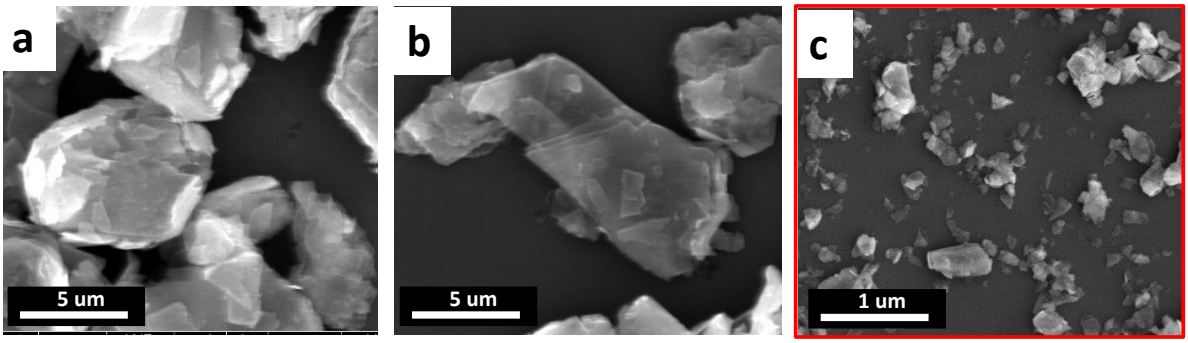

Figure S4. SEM images of MoS_2 processed in the VFD at (a) 4k, (b) 6k rpm and (c) 8k rpm (optimised rotational speed) rotational speed, under continuous flow at a flow rate 0.45 mL.min^{-1}, tilt angle θ 45°, with the concentration of the as received MoS_2 at 5 mg. mL^{-1} (optimised concentration) dispersed at a 1:1:1 mixture of Ethanol/water/DMF.

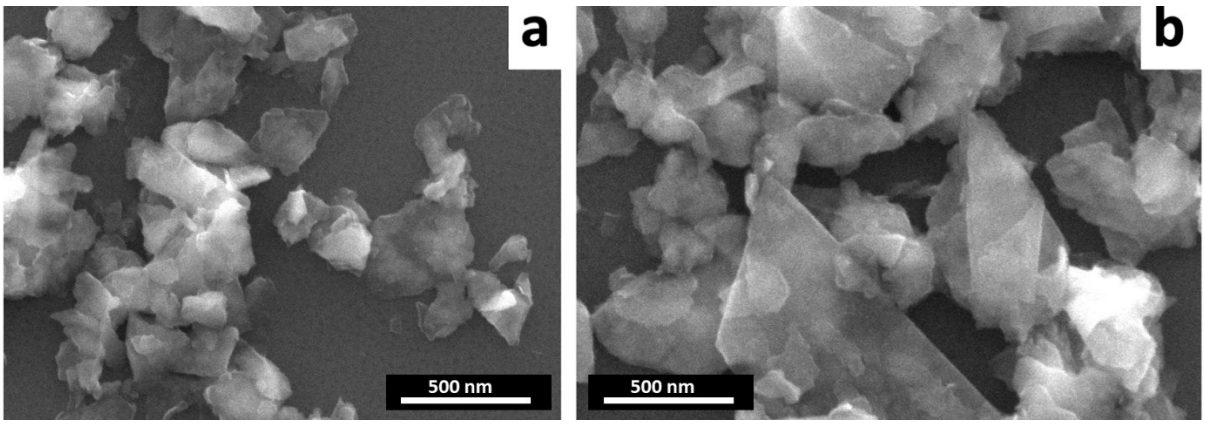

Figure S5. SEM images of MoS_2 **(with different size of as received (bulk) materials)** processed in the VFD at 8k rpm rotational speed, under confined mode for 30 mins, tilt angle θ 45°, with a concentration of 5 mg. mL^{-1} (optimised concentration).

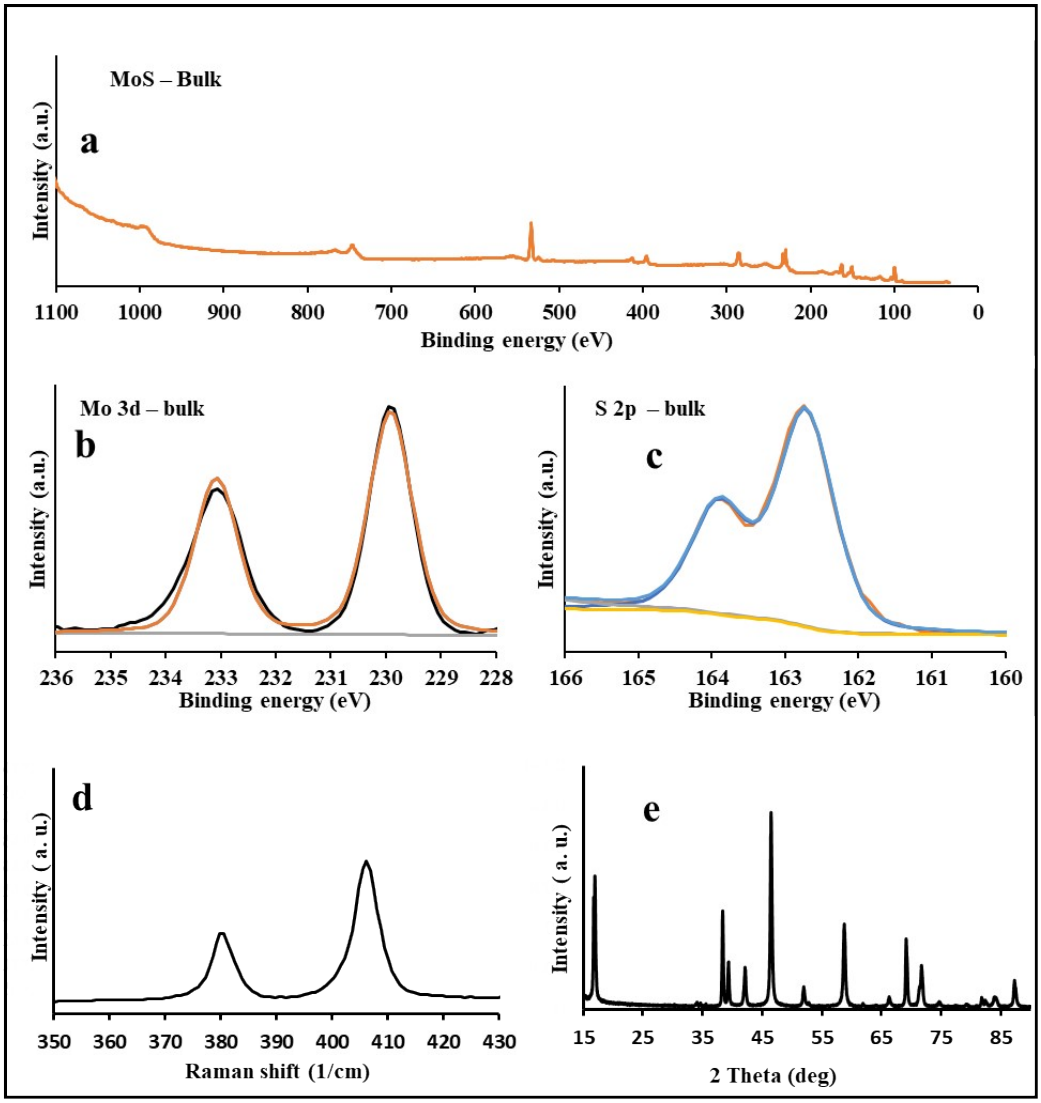

Figure S6. (a)Survey XPS, (b-c) High-resolution spectra of Mo 3d and S 2p (d) Raman spectra and (e) XRD patterns for bulk MoS_2.

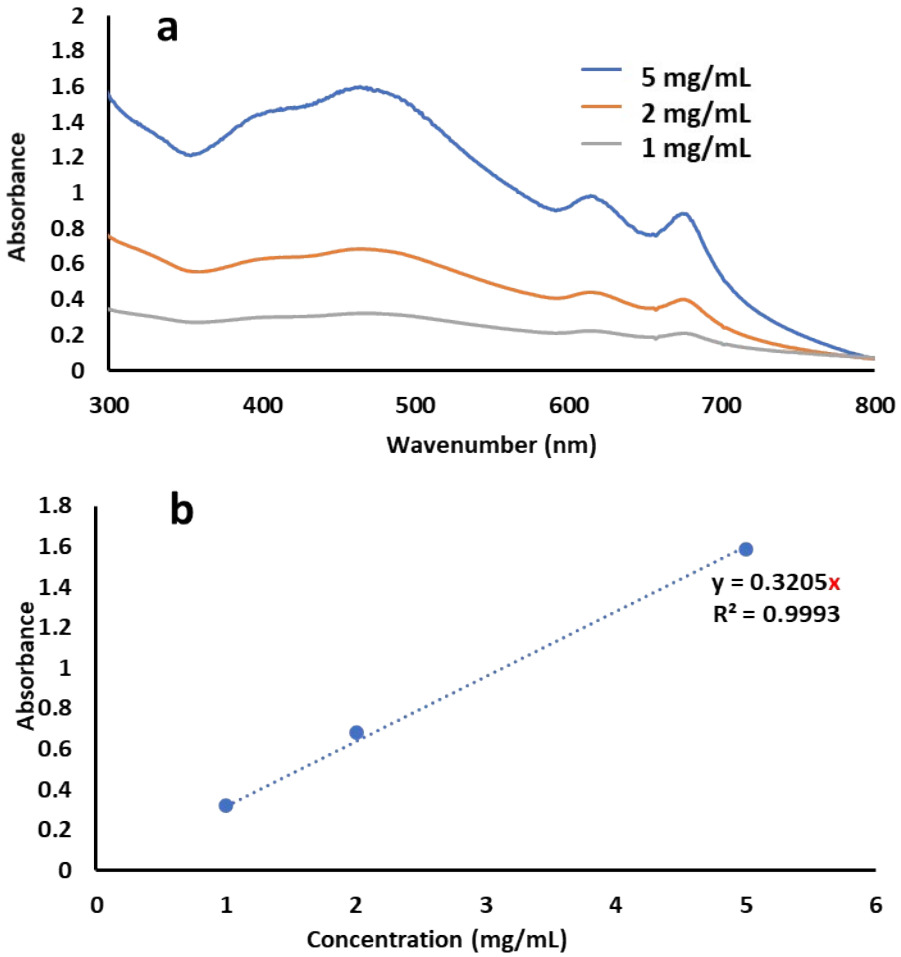

Figure S7. (a) UV-vis absorption spectra and (b) the corresponding calibration curves for exfoliated MoS2 nanosheets using VFD under optimised conditions.

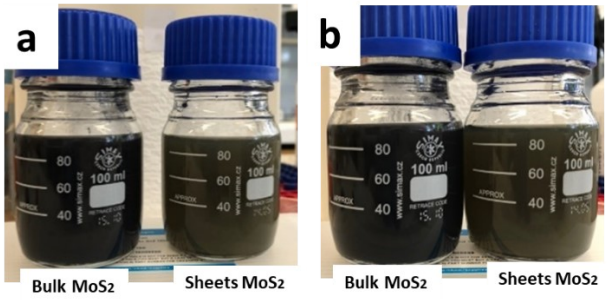

Figure S8. Photographs of the bulk MoS_2(left) and exfoliated MoS_2 (right) directly after processed in the VFD under continuous flow (a) and after 3 months on the bench (b)

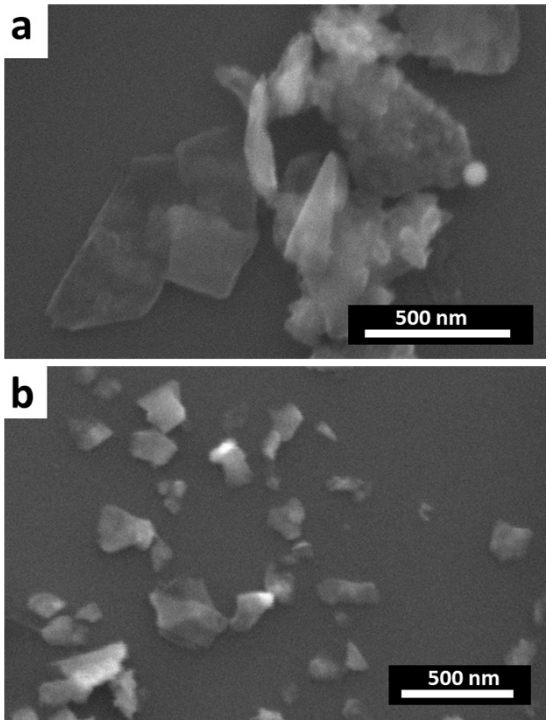

Figure S9. SEM images of exfoliated MoS_2 nanosheets directly after processed in the VFD under continuous flow at optimised conditions (a) and after 3 months on the bench (b)