Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Solvo-thermal microwave-powered two-dimensional materials exfoliation

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Experimental Section

Boron nitride (BN) was purchased from Momentive, Inc., graphite from Asbury Graphite Mills, Inc. MoS2 from SPI Supplies, Inc. The black phosphorus (BP) with high purity (99.998%) was purchased from Smart Elements, Inc. N-Methyl-2-pyrrolidone (NMP), Isopropyl alcohol (IPA), Dimethylformamide (DMF) were purchased from Sigma-Aldrich separately.

A bench-mixer (Benchmark Scientific, Inc.) was used bulk TDMs pre-selectivity. 200mg micron-sized TDMs raw material was added into 15ml deionized water, and stirred by bench-mixer for 1min at 3000 rpm. Then, the solution was centrifuged at 500 rpm for 15min. After centrifugation, the solvent was removed by pipette, and the precipitation was further dried in vacuum oven overnight for further microwave application.

To begin with, around 50 mg TDMs were glue bonded onto SiO2/Si wafer, followed by a few times gentle tapping, to ensure TDMs uniformly bonded on SiO2/Si wafer. The bulk TDMs samples glued on wafer were together transferred into organic solution and heated by microwave oven (Panasonic) (2.54GHz, 125kW) in the surrounding of organic solvent for sufficient time (depend on each materials and their matching solvents).

The afore-prepared TDMs on solution was immediately transferred and heated into the microwave oven. The power output was set to be 125W, which is the 1/10 the Microwave oven maximum power level, after sufficient microwave exfoliation (empirically 2 minutes), the SiO2/Si wafer is then taken out to dry for further characterization.

Microwave synthesis protocol

Boron nitride (BN) was purchased from Momentive, Inc., graphite from Asbury Graphite Mills, Inc. MoS2 from SPI Supplies, Inc. The black phosphorus (BP) with high purity (99.998%) was

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Results quantifying the exfoliation of *black phosphorus*

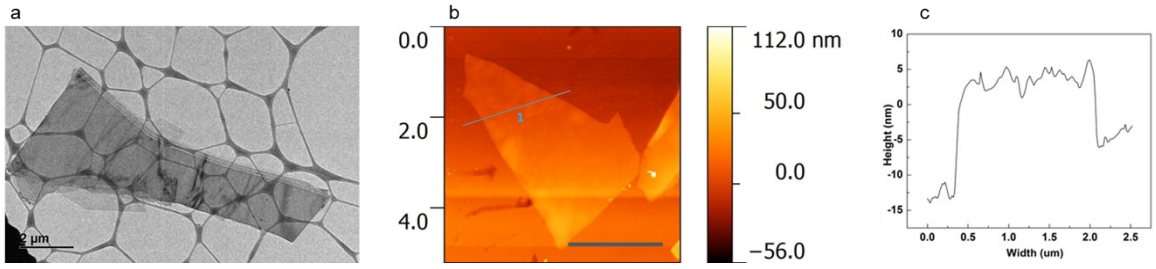

Figure S1. a) The morphological image of as exfoliated Black phosphorus (BP) from transmission electron microscopy; b) atomic force microscopy of each individual as exfoliated BP flakes; (scale bar: 2um) c) thickness profile of BP.

Steps in exfoliation of MoS_2

Figure S2. The in-situ characterization of MoS_2 edge non-rolling by optical microscopy: a) MoS2 surface structure after microwave 2 min; b) 6 min; c) 10 min (scale bar: 100 um).