Supporting Information for

Salt-template-assisted synthesis of robust 3D honeycomb-like structured MoS₂ and its application as a lithium-ion battery anode

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Figure S1. (a)-(b) SEM images of the complex of $NH_2CSNH_2-(NH_4)_6Mo_7O_{24}/NaCl$ after freeze-drying process, showing that the NaCl particles uniformly coated with a thin film of $NH_2CSNH_2-(NH_4)_6Mo_7O_{24}$ complex were assembled into a 3D structure during the freeze-drying process. (c)-(d) SEM images of the 3D honeycomb-structured MoS₂ products before removing the NaCl, indicating that the 3D assembly was well preserved after the calcination process and the MoS₂ walls were actually formed between the surface of 3D NaCl assembly.



Figure S2. Selected area electron diffraction (SAED) pattern of 3D honeycomb-structured MoS_2 , radially demonstrating the characteristic diffraction rings of (100), (103), (105) and (110) planes belonging to polycrystalline MoS_2 , respectively.



Figure S3. Typical TEM images of 3D honeycomb-structured MoS₂ in the cases of (a)-(b) the faster freeze-drying process and (c)-(d) the slower freeze-drying process.



Figure S4. (a) Raman spectrum of 3D honeycomb-structured MoS₂. (b) Magnified Raman spectrum of 3D honeycomb-structured MoS₂, exhibiting the typical MoS₂ characteristic signature.



Figure S5. XPS spectra of (a) Mo3d and S2s peaks, and (b) S2p peaks of 3D honeycomb-structured MoS_2



Figure S6. (a) Nitrogen adsorption-desorption isotherms, and (b) pore size distribution curve of

3D honeycomb-structured MoS₂.



Figure S7. (a) Nitrogen adsorption–desorption isotherms, and (b) pore size distribution curve of 3D honeycomb-structured MoS₂ in the case of the faster freeze-drying process.



Figure S8. (a) SEM image of MoS_2 blocks synthesized by calcining the freeze-dried mixture of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and CH_4N_2S without adding NaCl at the same preparation conditions of 3D honeycomb-structured MoS_2 . (b) CV curves of MoS_2 blocks for the initial three cycles at a scanning rate of 0.1 mV s⁻¹ over a voltage window of 0.005-3 V.



Figure S9. (a) Cycling performance at the initial 25 cycles and (b) rate capability of the electrode

of 3D honeycomb-structured MoS₂ obtained by a faster freeze-drying process.



Figure S10. (a) Electrochemical impedance spectroscopy (EIS) with Randles equivalent circuit inset, and (b) linear fits of the relationship between Z' and $\omega^{-1/2}$ in the low-frequency region of 3D honeycomb-structured MoS₂, MoS₂ blocks and commercial MoS₂ powders after the rate test. (The larger solution resistance of the commercial MoS₂ powders electrode should be blamed for the tremendous dissolution into electrolyte of sulfur generated by the serious pulverization coupled with agglomeration of active materials during rate cycles, on account of the poor crystallinity and severe structural defects of commercial MoS₂.)



Figure S11. (a) Typical SEM image and (b) partially magnified SEM image of the anode of 3D honeycomb-like structured MoS₂ with being soaked in DMC for 6h to expose active materials after the rate test, demonstrating a good achievement of 3D porous structure free from re-stacking even after the rapid charge/discharge process.



Figure S12. (a) STEM image of a magnified MoS₂ nanosheet taken from a representative 3D honeycomb-like structured MoS₂ after rate performance. EDX element mappings of (b) Mo and (c) S, indicating the homogeneous distribution of Mo and S elements in 3D honeycomb-structured MoS₂, even after the rapid lithiation/delithiation rate cycles.