Electronic Supplementary Information for

Facile synthesis of MoS_2@CMK-3 nanocomposite as an improved anode material for lithium-ion batteries

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Experimental Section

Synthesis of SBA-15: 4.0 g of Pluronic P123 ($EO_{20}PO_{70}EO_{20}$, Aldrich) was dissolved in 30 g of deionized (DI) water and 120 g of HCl (2M). Then 8.5 g of tetraethoxysilane (TEOS, Xilong Chemical Co., Ltd.) was added into the solution. The mixture was vigorously stirred at 35 ^oC for 24 h, followed by hydrothermal treatment at 100 ^oC for 24 h in two 100 mL Teflon-lined autoclaves. The solid product was filtered, washed, dried, and finally calcined at 550 ^oC for 4 h in air.[1]

Synthesis of CMK-3: 1.25 g of sucrose (Sinopharm Chemical Reagent Co., Ltd.) was dissolved in 5 mL of DI water containing 0.14 g of concentrated H_2SO_4. Then 1.0 g of SBA-15 was dispersed in the solution by sonicating for 1 h, followed by heat treatment at 100 ^oC for 6 h and subsequently at 160 ^oC for 6 h. The impregnation procedures were repeated once with another 5 mL aqueous solution containing 0.8 g of sucrose and 0.09 g of concentrated H_2SO_4. The obtained dark brown composite was completely carbonized at 900 ^oC for 5 h in an argon flow. To remove the SBA-15 template, the resulting dark composite was stirred in a 5% HF water/ethanol solution at room temperature for 8 h.[2-4]

Synthesis of MoS_2@CMK-3 nanocomposite: 0.6 g of CMK-3 powders were firstly dispersed in 60 mL of concentrated HNO_3 and stirred for 1 h at 80 ^oC to induce hydrophilicity. Secondly, 0.5 g of ammonium tetrathiomolybdate (NH_4MoS_4) was dissolved in 25 mL of DI water at 100 ^oC. Then 0.1 g of CMK-3 was added into the solution, which was stirred for 1 h. The mixture was dried statically, and grounded to powder. The achieved powder was thermally decomposed in a tube furnace at 450 ^oC for 6 h under H_2/Ar (5:95 v/v) atmosphere with a heating rate of 5 $^oC\ min^{-1}$ to form the MoS_2@CMK-3 nanocomposite. For comparison, bulk MoS_2 was prepared by thermal decomposition of NH_4MoS_4 in a tube furnace at 450 ^oC for 6 h under H_2/Ar (5:95 v/v) atmosphere with a heating rate of 5 $^oC\ min^{-1}$.[5]

Structural and Electrochemical Characterizations: SEM measurements were conducted on a Hitachi S-4800 field emission scanning electron microscope operated at 15 kV. TEM and HRTEM analysis was performed on a Tecnai G2 F20 U-TWIN field emission transmission electron microscope operated at 200 kV. Thermogravimetric (TG) analysis was carried out on a TA-Q50 instrument. XPS spectra were recorded on an ESCALab220i-XL electron spectrometer from VG Scientific using 300W AlKα radiation. XRD measurements were conducted on a Rigaku D/max2500 diffractometer using CuKα radiation. Nitrogen adsorption and desorption isotherms at 77.3 K were carried out with a Nova 2000e surface area-pore size analyzer. Electrochemical experiments were performed using Swagelok-type cells. To prepare working electrodes, MoS_2@CMK-3, Super-P carbon black, and poly(vinylidene fluoride) (PVDF) with mass ratio of 80:10:10 were mixed into a homogeneous slurry with mortar and pestle, and then the obtained slurry was pasted onto pure Cu foil (99.9 %, Goodfellow). The electrolyte was 1 M $LiPF_6$ in EC/DMC/DEC (1:1:1 v/v/v) (Tianjing Jinniu Power Sources Material Co., Ltd.). Glass fibers (GF/D) from Whatman were used as separators and pure lithium metal foil (Aldrich) was used as the counter electrode. The Swagelok-type cells were assembled in an argon-filled glove box. Cyclic voltammetry was conducted on a Voltalab 80 electrochemical workstation at a scan rate of 0.1 mV s^{-1}. The discharge and charge measurements of the batteries were performed on an Arbin BT2000 system in the fixed voltage window between 5 mV and 3 V at room temperature. Electrochemical impedance spectral measurements were carried out with a PARSTAT 2273 advanced electrochemical system over the frequency range from 100 kHz to 100 mHz.

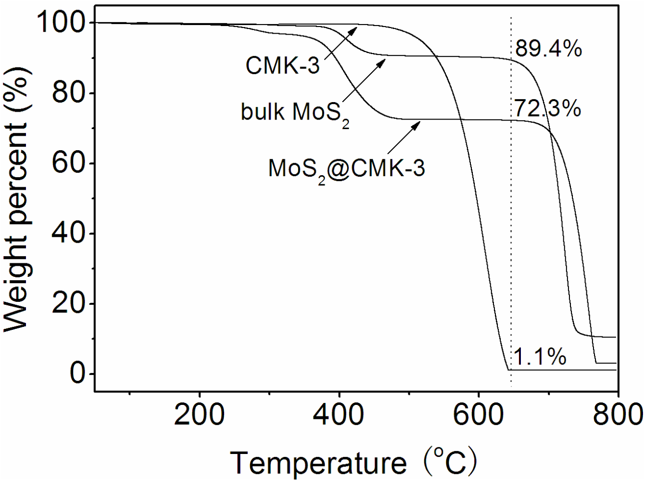

Figure S1. TG analysis curves of CMK-3, bulk MoS_2, and MoS_2@CMK-3 under air atmosphere at a heating rate of 10 °C min^{-1}. The residual weight percents of CMK-3, bulk MoS_2, and MoS_2@CMK-3 at 650 °C are collected to calculate the content of MoS_2 in the MoS_2@CMK-3 nanocomposite on the basis of the equation $W_{MoS2}\ X_{MoS2} + W_{CMK\text{-}3}\ (1\text{-}X_{MoS2}) = W_{MoS2@CMK\text{-}3}$, where X_{MoS2} is the content of MoS_2 in the MoS_2@CMK-3 and W_{MoS2}, $W_{CMK\text{-}3}$, and $W_{MoS2@CMK\text{-}3}$ are residual weight percents of bulk MoS_2, CMK-3, MoS_2@CMK-3. The content of MoS_2 in the MoS_2@CMK-3 is calculated to be approximately 80.6 wt%.

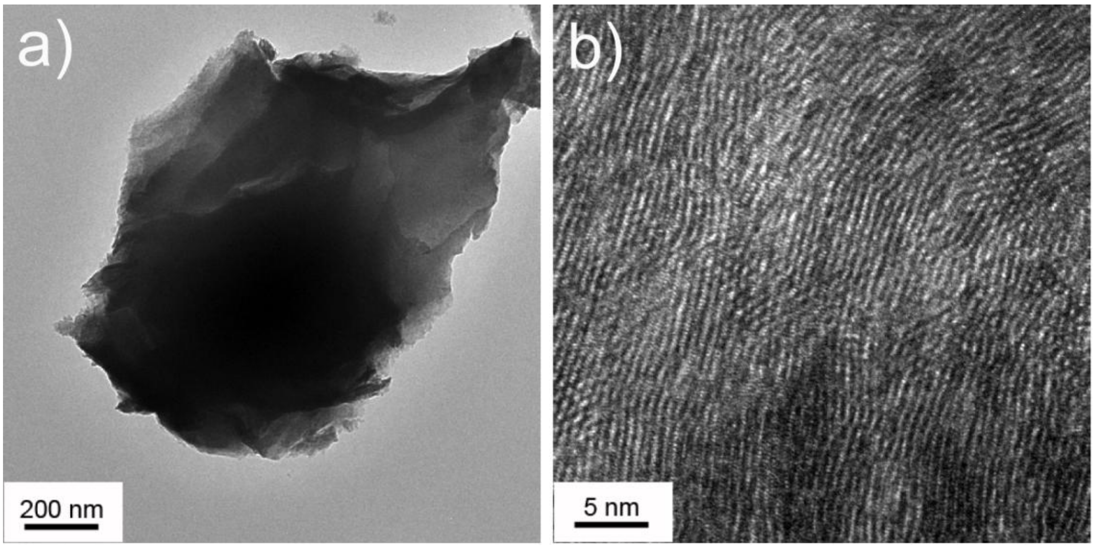

Figure S2. a) TEM image of bulk MoS_2, b) HRTEM image of bulk MoS_2.

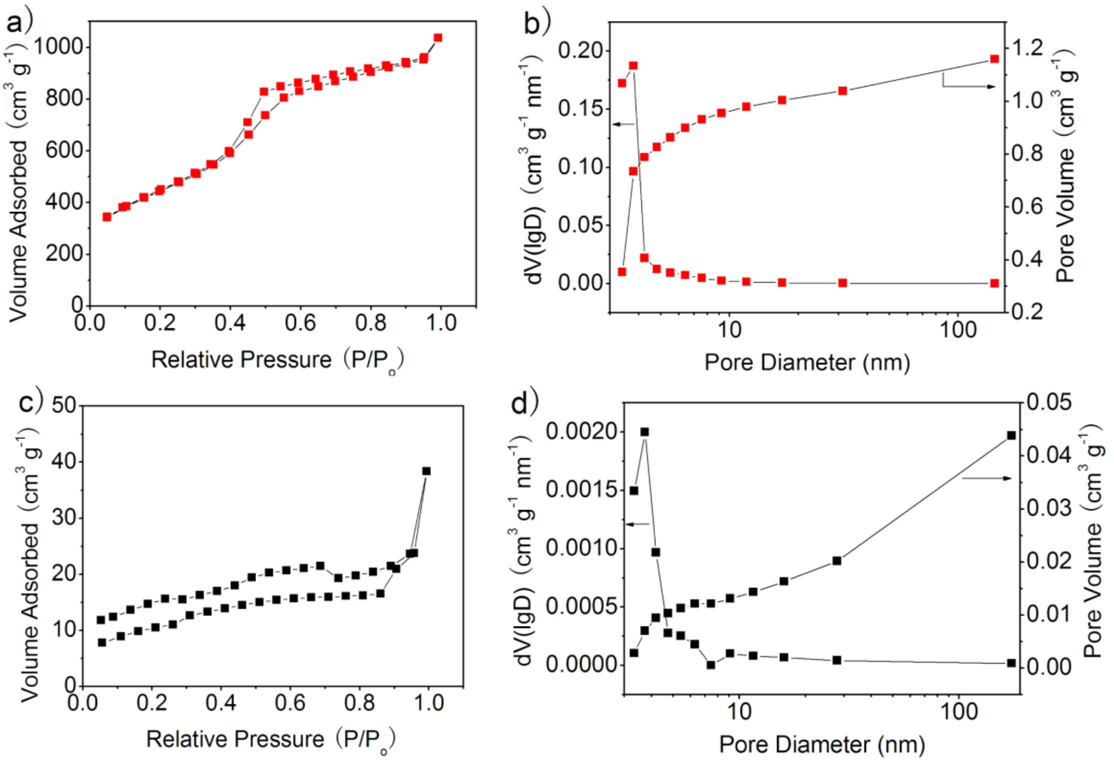

Figure S3. a), b) Nitrogen adsorption/desorption isotherms and pore-size distribution plot of CMK-3. c), d) Nitrogen adsorption/desorption isotherms and pore-size distribution plot of MoS_2@CMK-3.

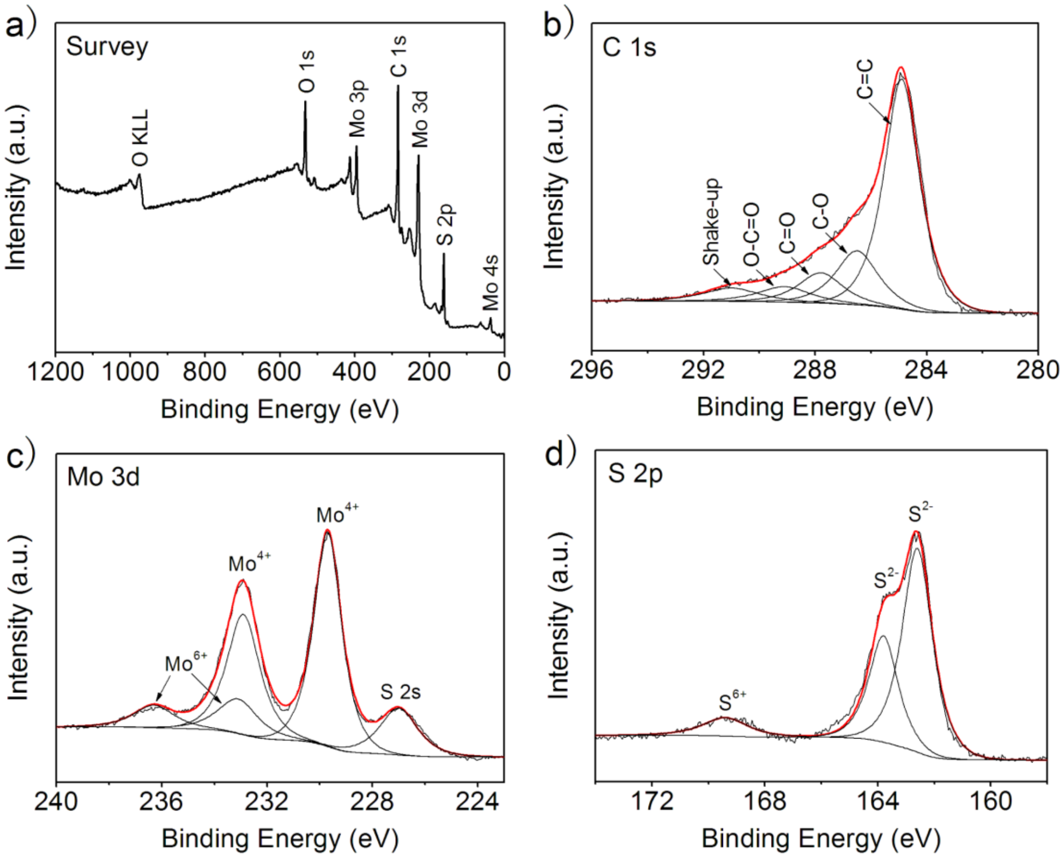

Figure S4. XPS of MoS_2@CMK-3 nanocomposite. a) Survey spectrum, b) C 1s spectrum, c) Mo 3d spectrum, and d) S 2p spectrum. The strong Mo $3d_{5/2}$ and S $2p_{3/2}$ bands at 229.7 and 162.6 eV, respectively, indicating that Mo^{4+} and S^{2-} are the dominant states. Small sub-bands of Mo $3d_{5/2}$ and Mo $3d_{3/2}$ at 233.1 eV and 236.3 eV are identified, suggesting the presence of Mo^{6+}, which might arise from MoO_3 owing to surface oxidation. In addition, the sub-band of S 2p at 169.4 eV is also observed, suggesting the existence of S^{6+}, which might derive from the residual SO_4^{2-} in the CMK-3.

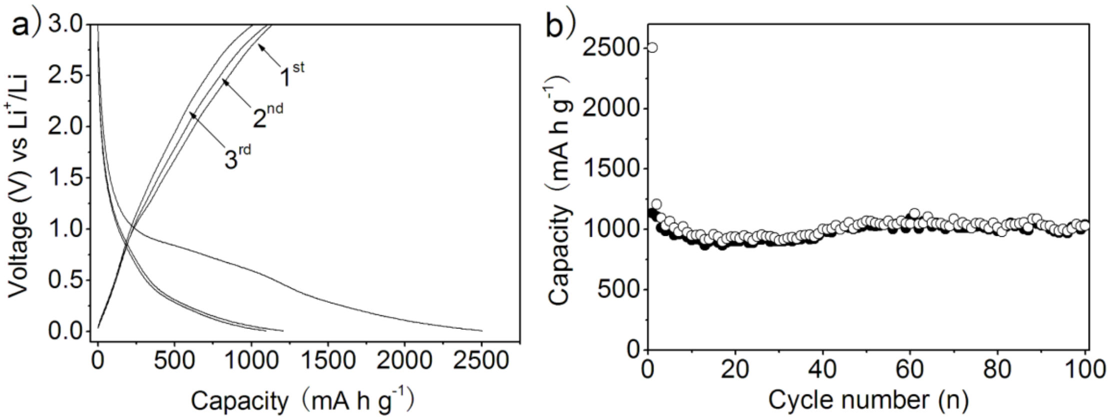

Figure S5. a) Galvanostatic discharge-charge profiles of the first three cycles and b) cycling performance of CMK-3 electrode under a current density of 250 mA g^{-1} within the voltage range of 0.005–3 V vs Li^+/Li.

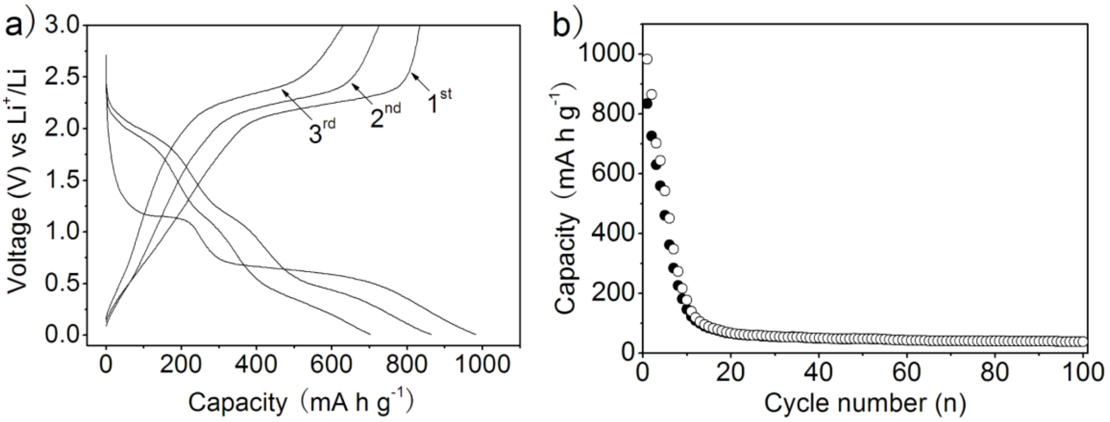

Figure S6. a) Galvanostatic discharge-charge profiles of the first three cycles and b) cycling performance of bulk MoS_2 electrode under a current density of 250 mA g^{-1} within the voltage range of 0.005–3 V vs Li^+/Li,.

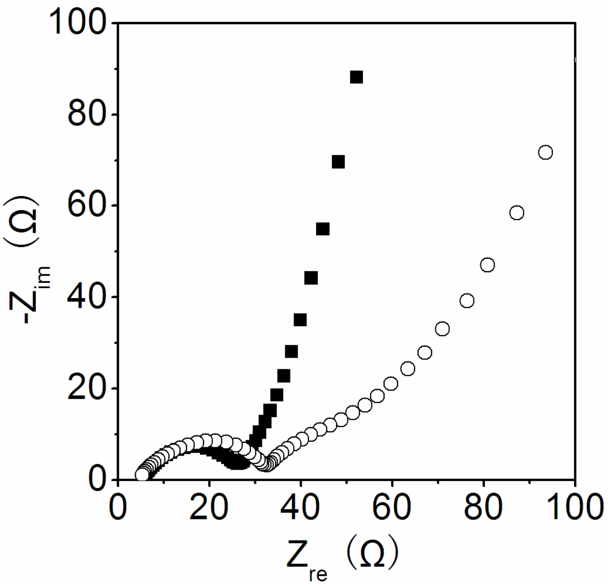

Figure S7. Nyquist plots of MoS_2@CMK-3 electrode before cycling (solid squares) and after 70 cycles (empty cycles).

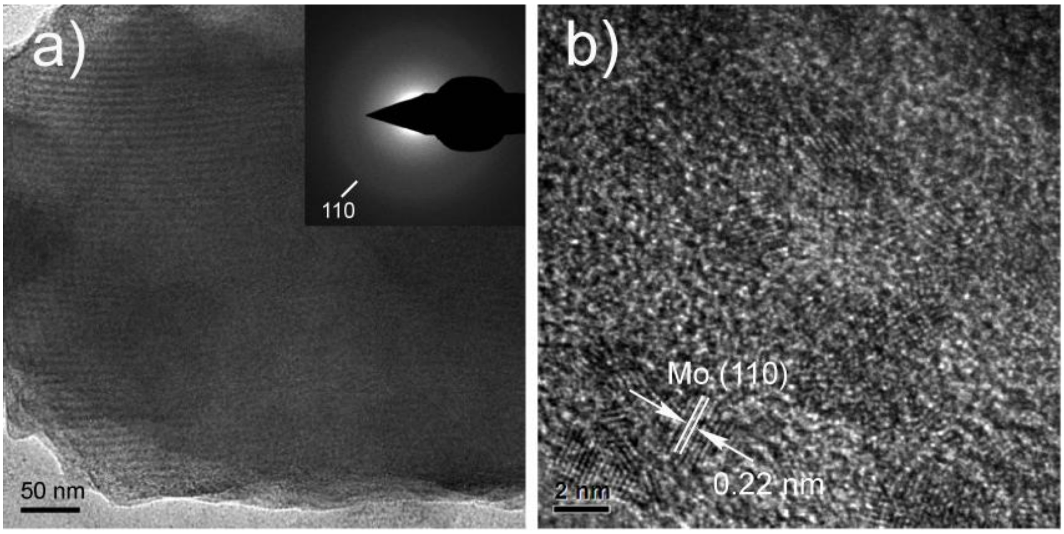

Figure S8. a), b) TEM and HRTEM images of MoS_2@CMK-3 after cycling 70 cycles, and the inset showing the corresponding SAED pattern.

References:

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