Electrochemical properties of SnO_2 nanoparticles immobilized within a metal-organic framework as anode material for lithium-ion battery

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Experimental Detals

Synthesis

1. MIL-101(Cr). A mixture of 1.86 g $CrCl_3 \cdot 6H_2O$, 1.16 g terephthalic acid (BDC), 50 ml H_2O was loaded into a 100 ml autoclave with a Teflon cup and heated at 210 °C for 24 h. Upon cooling down, the green suspension of nano MIL-101(Cr) was centrifuged and washed with hot DMF and EtOH. Then it was dispersed in NaOH solution (pH=12, 20 ml) and stirred overnight at 60 °C. After it was centrifuged and washed with deionized water, MIL-101(Cr) was obtained by drying at 120 °C in vacuum.

2. SnO_2@MIL-101(Cr). 0.2 g MIL-101(Cr) and 1.0 g $SnCl_4 \cdot 5H_2O$ were dispersed in 15 ml H_2O and stirred for 24 h in room temperature. Then it was centrifuged and washed once with 10 ml H_2O. After that it was dispersed in 100 ml NaOH solution (pH=12) and stirred over night at room temperature. Then the suspension was centrifuged and washed with deionized water (four times, 20 ml each time) to remove free ions in MIL-101(Cr). After drying at 180 °C in vacuum, SnO_2@MIL-101(Cr) was obtained. Bare SnO_2 was synthesized following the similar procedure in the absence of MIL-101(Cr) by adding NaOH solution into $SnCl_4$ solution.

3. Battery assembling. SnO_2@MIL-101(Cr) (bare SnO_2) was mixed with 10% acetylene black and 10% polyvinylidene fluoride (PVDF) in N-methyl pyrrolidone (NMP) separately. The slurry was coated on copper foil as anode (~0.3 mg-SnO_2/cm^2, whole carbon content 41.6 wt%). 2025 batteries were assembled with electrolyte of 1 M $LiPF_6$ in 1,3-dioxolan-2-one/dimethylcarbonate (EC/DMC, v:v=1:1), Celgard 2400 membrane and lithium foil counter electrode.

Characterization

PXRD patterns were recorded on a PANalytical X'Pert PRO diffractometer at 40 kV, 25 mA for Cu Kα, (λ=1.541 Å). SEM morphologies were investigated using a Hitachi S4800 field-emission scanning electron microscopy. TEM morphologies and EDS mappings were taken on a Hitachi HT7700 transmission electron microscopy. ICP-MS was performed on a Thermo Scientific XSERIES 2 ICP-MS system to determine SnO_2 content in SnO_2@MIL-101(Cr). The FTIR spectra were measured with a Nicolet Thermo Scientific Nicolet iS10 spectrometer. N_2 sorption properties were studied with a Quantachrome 20-E high speed gas sorption analyzer. The CV curves were collected with an Arbin electrochemical workstation at a scan rate of 0.1 mV s^{-1} between 0.02 and 2.5 V. The EIS data were collected with an Arbin electrochemical workstation. The charge/discharge profiles, cyclability, ratecapability and Coulombic efficiency were recorded with

a LAND battery cycler between 0.02 and 2.5 V.

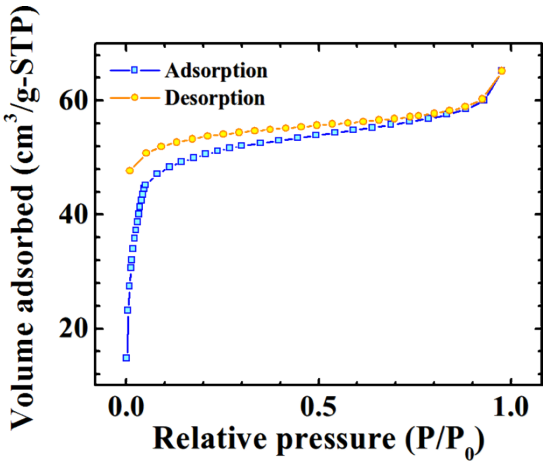

Fig. S1. N_2 adsorption/desorption isothermal of SnO_2@MIL-101(Cr) at 77 K.

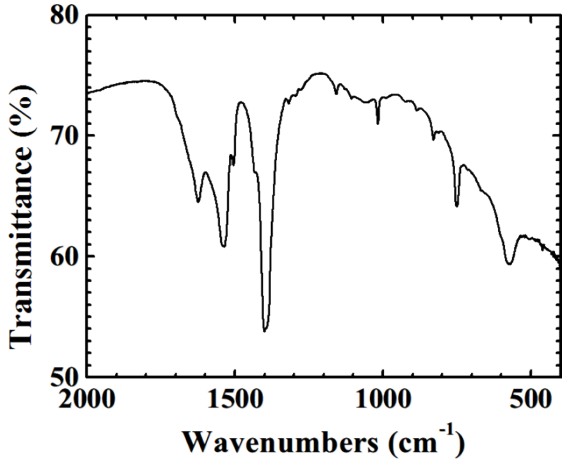

Fig. S2. FTIR spectra of SnO_2@MIL-101(Cr).

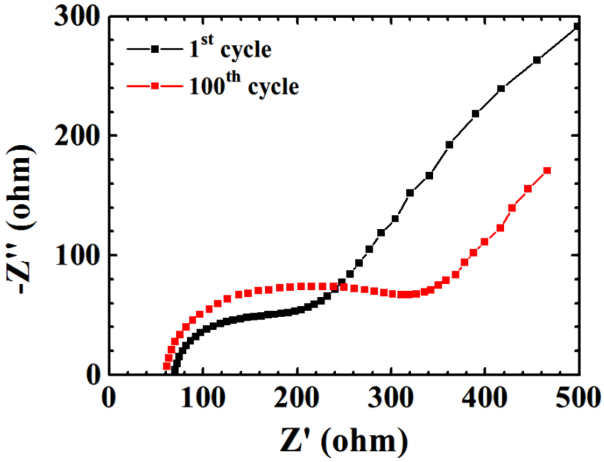

Fig. S3. EIS spectra for the half cell with SnO_2@MIL-101(Cr) anode after 1st cycle and 100th cycle.

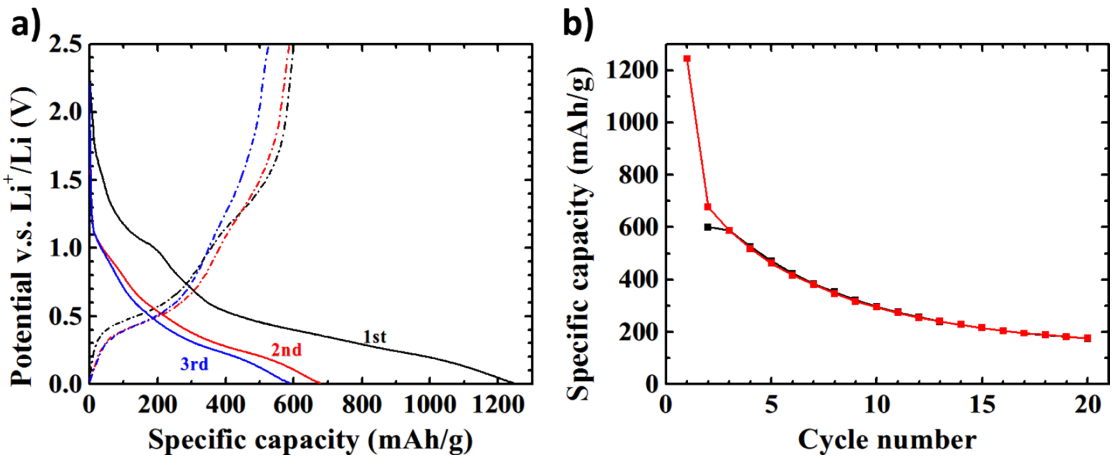

Fig. S4. (a) Galvanostatic charge/discharge profiles and (b) cycle performance at 0.1 C of bare SnO_2.