Supporting Information

Hierarchical $MoSe_2$ yolk-shell microspheres with superior Na-ion storage properties

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Experimental

Synthesis of yolk-shell structured MoO_3 and $MoSe_2$ microspheres

The yolk-shell structured MoO_3 microspheres were fabricated by spray pyrolysis and a subsequent combustion process, as described in our previous report.[42] For the selenization of the yolk-shell structured MoO_3 microspheres, selenium powders were used as the Se source. The MoO_3 microspheres and selenium powders were loaded in an alumina boat with a cover and placed in a quartz tube reactor, which was heated to 300°C for 24 h under a flow of 10% H_2/Ar mixture gas.

Characterization

The morphologies of the MoO_3 and $MoSe_2$ samples were investigated by field-emission scanning electron microscopy (FE-SEM; S-4800, Hitachi) and transmission electron microscopy (FE-TEM; JEM-2100F, JEOL). The crystal structures of the powders were investigated by X-ray diffractometry (XRD; X'Pert PRO MPD) using Cu Kα radiation (λ = 1.5418 Å) at the Korea Basic Science Institute (Daegu). The surface areas of the powders were measured by the Brunauer–Emmett–Teller (BET) method using N_2 as the adsorbate gas. The densities of the powders were measured by mercury porosimeter (Auto Pore IV 9500).

Electrochemical Measurements

The electrochemical properties of the MoO_3 and $MoSe_2$ samples were analyzed in a 2032-type coin cell. The anode was prepared from a mixture of the active material, carbon black, and sodium carboxymethyl cellulose (CMC) in a weight ratio of 7:2:1. Na metal and a microporous polypropylene film were used as the counter electrode and separator, respectively. The electrolyte was a solution of 1 M $NaClO_4$ (Aldrich) in a 1:1 volume mixture of ethylene carbonate/dimethyl carbonate (EC/DMC) with 5 wt% fluoroethylene carbonate. The discharge and charge characteristics of the samples were investigated by cycling in the voltage range 0.001–3 V at various current densities. The cyclic voltammograms (CVs) were recorded at a scan rate of 0.1 mV s^{-1}. Electrochemical impedance spectra were obtained by AC electrochemical impedance spectroscopy (EIS) with a ZIVE SP1 over a frequency range of 0.01 Hz–100 kHz and at the potential amplitude of 10 mV.

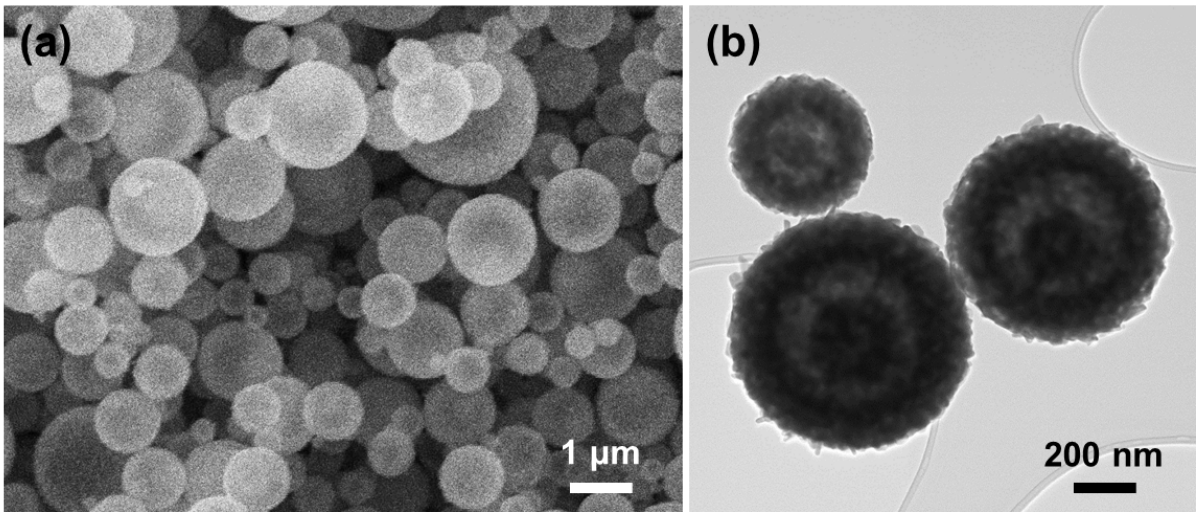
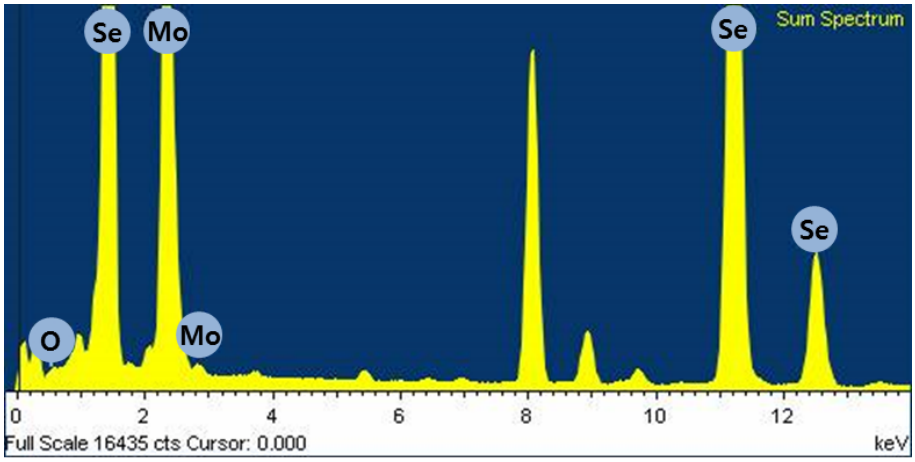


Fig. S1 Morphologies of the yolk-shell-structured MoO_3 microspheres: (a) SEM image and (b) TEM image.



Element	Atomic %
Mo	34.36
Se	63.94
O	1.7

Fig. S2 TEM-energy dispersive X-ray (EDX) spectrum of the hierarchical $MoSe_2$ yolk-shell microspheres.

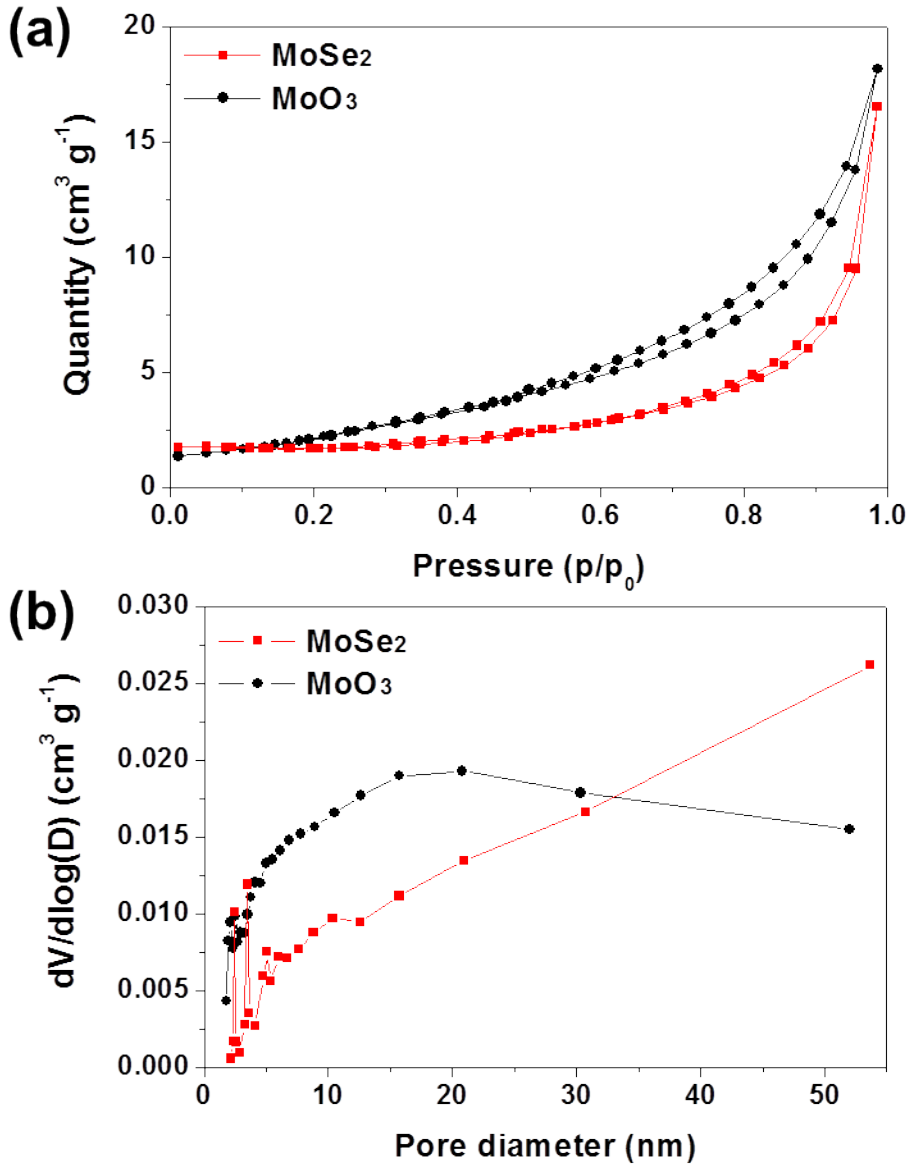


Fig. S3 (a) Nitrogen adsorption-desorption isotherms and (b) pore size distribution curves of the yolk-shell-structured $MoSe_2$ and MoO_3 microspheres.

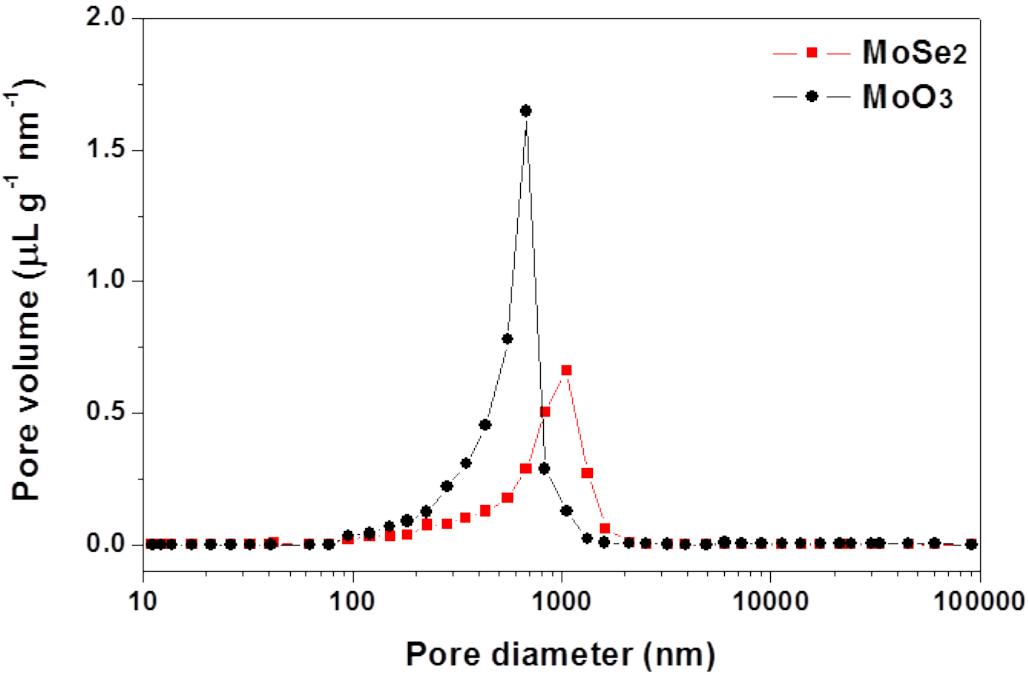


Fig. S4 Pore size distributions determined by mercury porosimetry of the yolk-shell-structured $MoSe_2$ and MoO_3 microspheres.

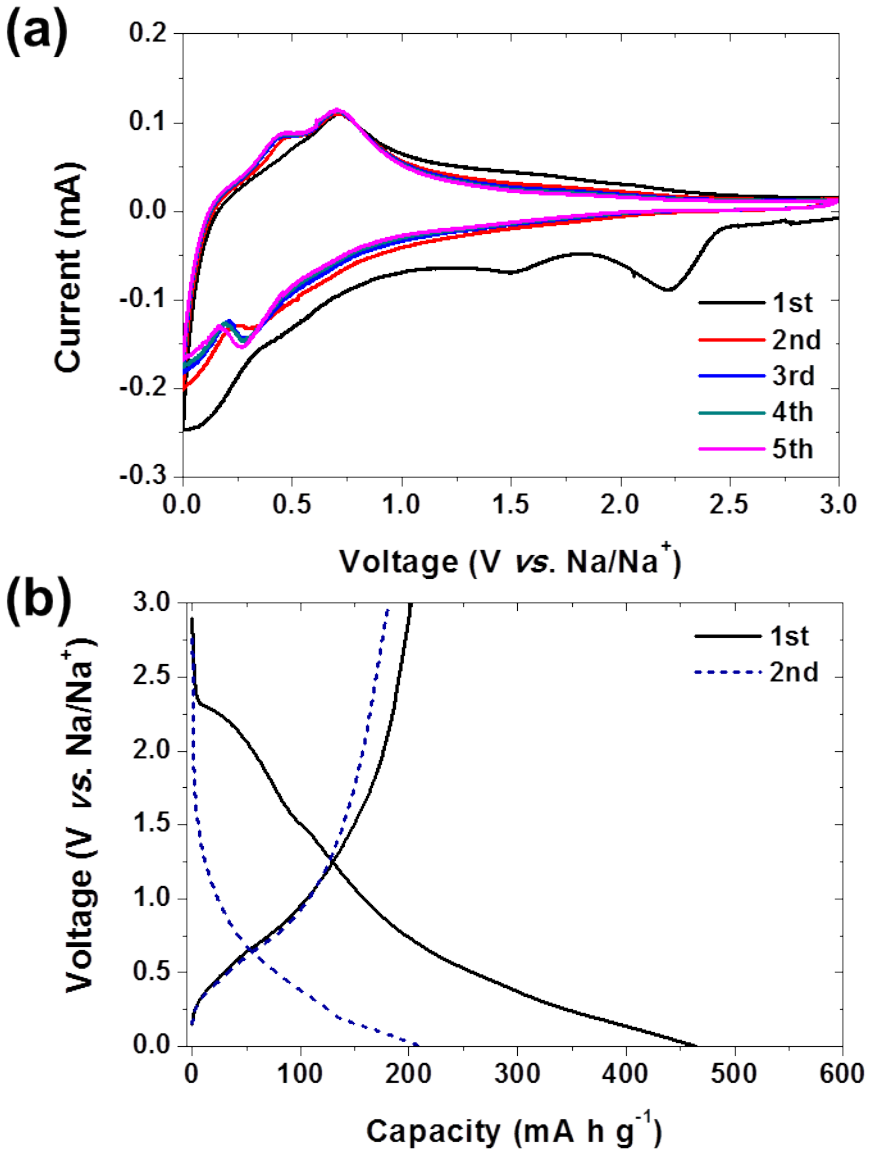


Fig. S5 Electrochemical Na-ion insertion and extraction behaviors of the yolk-shell-structured MoO_3 microspheres: (a) cyclic voltammograms and (b) discharge/charge profiles.

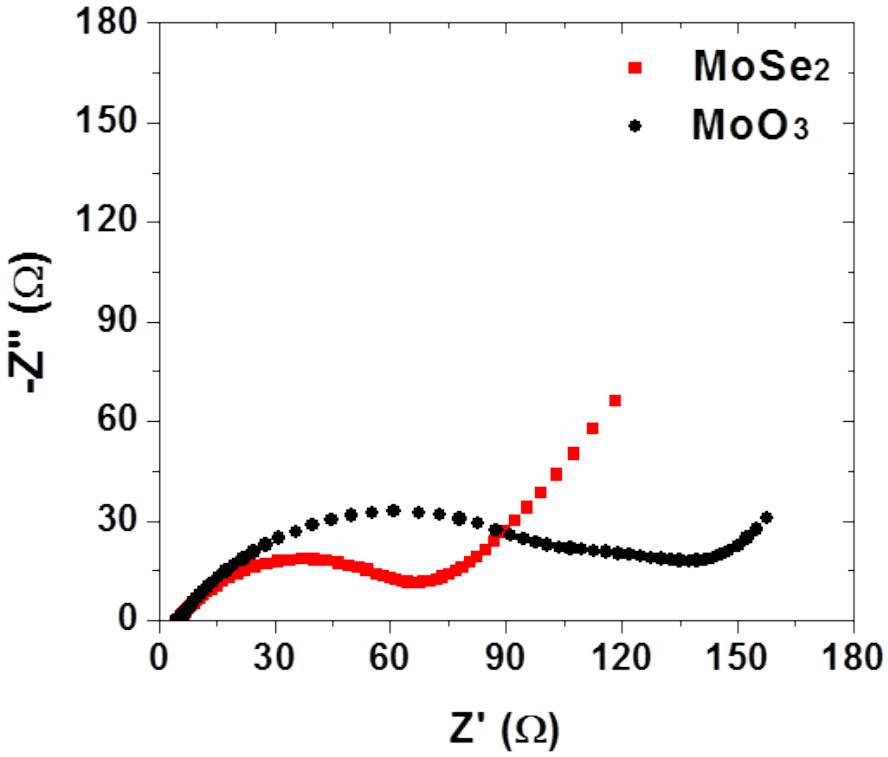


Fig. S6 Nyquist plots of the electrochemical impedance spectra after cycling.