Supporting Information

Facile one-pot synthesis of spherical zinc sulfide–carbon nanocomposite powders with superior electrochemical properties as anode materials for Li-ion batteries

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This file includes:

- Detailed experimental procedure.
- (a) XRD patterns of the powders prepared from the spray solutions with and without sucrose. (b) and (c) TG/DSC curves of the bare ZnS and ZnS-C composite powders prepared from the spray solutions with and without sucrose. (d) N₂ absorption-desorption isotherms for the ZnS-C composite powders.
- TEM and dot-mapping images of the bare ZnS powders prepared from the spray solution without sucrose.
- SEM images of the bare ZnS and ZnS-C composite powders after 300 cycles.
- SEM images of the ZnS-C powders.
- N₂ absorption-desorption isotherms for the bare ZnS and ZnS-C composite powders.
- TG/DSC curves of the ZnS-C composite powders.
- Cycle voltammograms of the ZnS-C composite powders.
- TEM and dot-mapping images of bare ZnS and ZnS-C nanocomposite powders.

Detailed experimental procedure:

ZnS–C nanocomposite powder was prepared directly by the spray pyrolysis of a solution containing Zn, S, and C components. The spray pyrolysis system consisted of a droplet generator, quartz reactor, and powder collector. A 1.7-MHz ultrasonic spray generator with six vibrators was used to generate a large quantity of droplets, which were transported to a high-temperature tubular reactor using a N₂ carrier gas flowing at 5 L min-1. The droplets evaporated, decomposed, and/or crystallized in the quartz reactor. The length and diameter of the quartz reactor were 1000 and 55 mm, respectively, and the temperature inside the reactor was fixed at 800°C. The spray solution was prepared by dissolving zinc nitrate (Zn(NO₃)₂, Junsei) and thiourea ((NH₂)₂CS, Junsei) in a ratio of 1:2 in distilled water. The total concentration of Zn and S components was 0.25 M. The concentration of sucrose, which was used as the C source, was varied from 0.05 to 0.3 M.

The crystal structures of the composite powders were investigated using X-ray diffractometry (XRD, Rigaku DMAX-33). The morphology of the composite powders was observed using scanning electron microscopy (SEM, JEOL JSM-6060) and transmission electron microscopy (TEM, JEOL, JEM-2010). The surface areas of the powders were measured by employing the Brunauer–Emmett–Teller (BET) method with N₂ as the adsorbate gas.

The capacities and cycling properties of anodes made from the composite powders were measured using 2032-type coin cells. The anodes were prepared by mixing 28 mg of composite powder, 6 mg of carbon black, and 6 mg of sodium carboxymethyl cellulose (CMC) in distilled water. Li metal was used as the counter electrode and polypropylene film was used as the separator. The electrolyte was 1 M LiPF₆ mixed in a 1:1 volume ratio with ethylene carbonate/dimethyl carbonate (EC/DMC) with 2% vinylene carbonate. The charge/discharge characteristics of the samples were measured at room temperature and under various current densities over the voltage range of 0.01-3.0 V. Cyclic voltammetry measurements were carried out at a scan rate of 0.1 mV s-1

Properties of the ZnS-C powders:



Fig. S1 (a) XRD patterns of the powders prepared from the spray solutions with and without sucrose. (b) and (c) TG/DSC curves of the bare ZnS and ZnS-C composite powders prepared from the spray solutions with and without sucrose. (d) N_2 absorption-desorption isotherms for the ZnS-C composite powders.

TEM and dot-mapping images of the bare ZnS powders:



Fig. S2 TEM and dot-mapping images of the bare ZnS powders prepared from the spray solution without sucrose.

SEM images of the powders after cycling:



Fig. S3 SEM images of the bare ZnS (a) and ZnS-C composite (b) powders after 300 cycles.

SEM images of the ZnS-C powders:



Fig. S4 SEM images of the ZnS-C powders prepared from the spray solutions with and without sucrose; (a) 0 M sucrose, (b) 0.05 M sucrose, (c) 0.1M sucrose, (d) 0.3M sucrose



N2 absorption-desorption isotherms for the bare ZnS and ZnS-C composite powders:

Fig. S5 N_2 absorption-desorption isotherms for the bare ZnS and ZnS-C composite powders prepared from the spray solutions with and without sucrose; (a) 0 M sucrose, (b) 0.05 M sucrose, (c) 0.3M sucrose.

TG/DSC curves of the ZnS-C composite powders:



Fig. S6 TG/DSC curves of the ZnS-C composite powders prepared from the spray solution with sucrose; (a) 0.05 M sucrose, (b) 0.3 M sucrose.

Cycle voltammograms of the ZnS-C composite powders:



Fig. S7 Cycle voltammograms of the ZnS-C composite powders for the first five cycles at a scan rate of 0.1 mV s^{-1} in the voltage range of 0.01-3V; (a) 0.05 M sucrose, (b) 0.3 M sucrose.



TEM and dot-mapping images of bare ZnS and ZnS-C nanocomposite powders:

Fig. S8 TEM and dot-mapping images of bare ZnS and ZnS-C nanocomposite powders prepared from the spray solutions with and without sucrose; (a) 0 M sucrose, (b) 0.05 M sucrose, (c) 0.1 M sucrose, (d) 0.3 M sucrose.