# Supporting Information

Micelles templated NiO hollow nanospheres as anode materials in lithium ion batteries

Synthesis of $PEO_{47}–b–PAA_{90}–b–PS_{80}$ via RAFT-Controlled Radical Polymerization.

Materials. Poly(ethylene oxide) (PEO) based chain transfer agent (PEO_{47}–CTA) was prepared as reported previously. 2,2'–Azobis(isobutyronitrile) (AIBN) was crystallized from methanol. Styrene was washed with an aqueous alkaline solution and distilled from calcium hydride under reduced pressure. Acrylic acid, *N,N*-dimethylformamide (DMF), and dioxane were dried over 4 Å molecular sieves and distilled under reduced pressure. Na_2CO_3 Wako), $CaCl_2$ (Katayama), tris buffer (Katayama), and naproxen sodium (Sigma-Aldrich) were used without further purification.
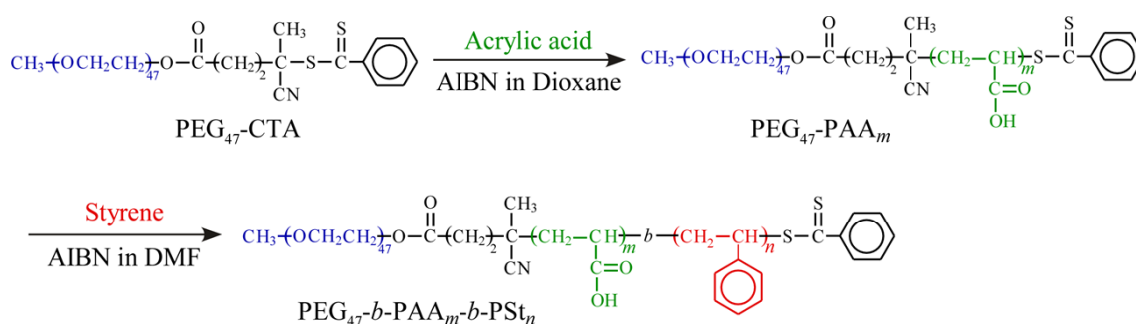

Scheme S1. Synthesis of PEG_{47}-*b*-PAA_m-*b*-PSt_n via RAFT-controlled radical polymerization.

Preparation of $PEG_{47}–b–PAA_m$. Acrylic acid (2.18 g, 30.3 mmol), AIBN (12.5 mg, 0.08 mmol), and PEO_{47}–CTA (475 mg, 0.20 mmol) were dissolved in dioxane (30 mL). The solution was degassed by purging with Ar gas for 30 min. Polymerization was carried out at 60 °C for 40 h. The polymerization mixture was dialyzed against pure water for one week. The diblock copolymer ($PEO_{47}–b–PAA_{90}$) was recovered by a freeze-drying technique (1.70 g, 64.0 %). Number average-degree of polymerization (DP) of PAA block was estimated from ^{1}H NMR spectrum in DMSO-d_6 to be 90. The number-average

molecular weight, M_n(NMR) for the block copolymer estimated from ^{1}H NMR is 8.85 x 10^3. M_n(GPC) and molecular weight distribution (M_w/M_n) were 1.53 x 10^4 and 1.31, respectively.

Preparation of PEO$_{47}$–*b*–PAA$_{90}$–*b*–PS$_{80}$. Styrene (10.4 mg, 10 mmol), AIBN (8.23 mg, 0.05 mmol), and PEO$_{47}$–*b*–PAA$_{90}$ (1.11 g, 0.13 mmol) were dissolved in DMF (100 mL). The solution was degassed by purging with Ar gas for 30 min. The polymerization was carried out at 60 °C for 24 h. The polymerization mixture was dialyzed against acetone for 3 days and pure water for one day. The obtained triblock copolymer (PEO$_{47}$–*b*–PAA$_{90}$–*b*–PS$_{80}$) was recovered by a freeze-drying technique (2.09 g, 18.2 %). DP of the PS block was 80 as estimated by ^{1}H NMR in DMSO-d_6. M_n(NMR) value for PEO$_{47}$–*b*– PAA$_{90}$–*b*–PS$_{80}$ is 1.88 x 10^4. M_n(GPC) and M_w/M_n were 9.34 x 10^3 and 1.22, respectively.

Measurements. Nuclear Magnetic Resonance (NMR). ^{1}H NMR spectra were obtained with a Bruker DRX-500 spectrometer operating at 500 MHz.
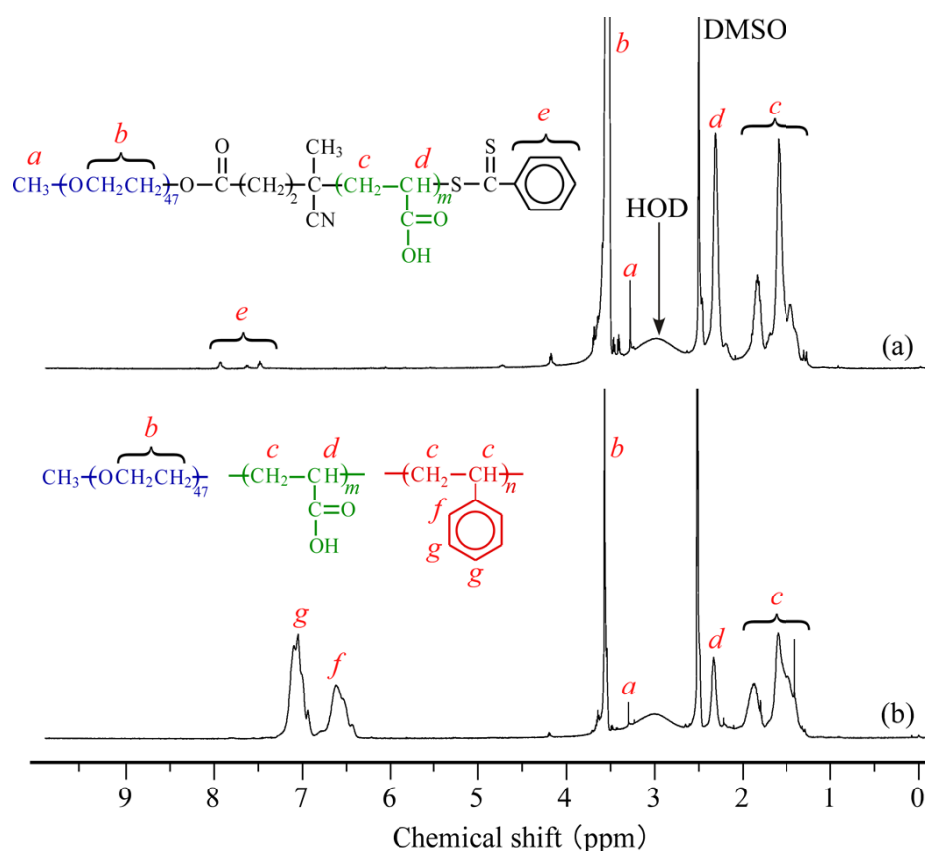
**Figure S2.** 500 MHz ^{1}H NMR spectra of (a) PEG$_{47}$-*b*-PAA$_{90}$ and (b) PEG$_{47}$-*b*-PAA$_{90}$-*b*-PSt$_{80}$ in DMSO-d_6 at 100 ºC with the corresponding assignments.

Gel-Permeation Chromatography (GPC). GPC measurements of PEO_{47}–b–PAA_{47} were performed using a refractive index (RI) detector equipped with a Shodex GF-7M HQ column working at 40 °C under a flow rate of 0.6 mL/min. A phosphate buffer (pH 8) containing 10 vol % acetonitrile was used as eluent. M_n and M_w/M_n for PEO_{47}–b–PAA_{47} were calibrated with standard sodium poly(styrenesulfonate) samples. GPC measurements of PEO_{47}–b–PAA_{90}–b–PS_{80} were performed at 40 °C with a Shodex DS-4 pump and an RI-101 refractive index detector using Shodex one KF-805L and three KF803L columns connected in series. THF was used as eluent at a flow rate of 1.0 mL/min. M_n and M_w/M_n were calibrated with standard polystyrene samples.
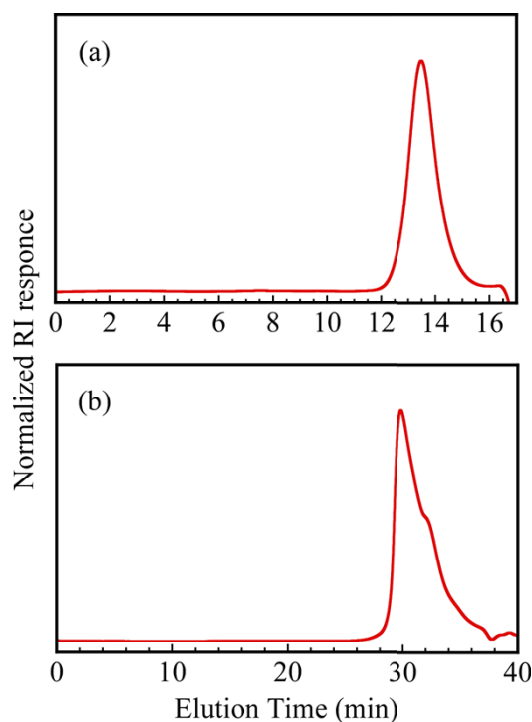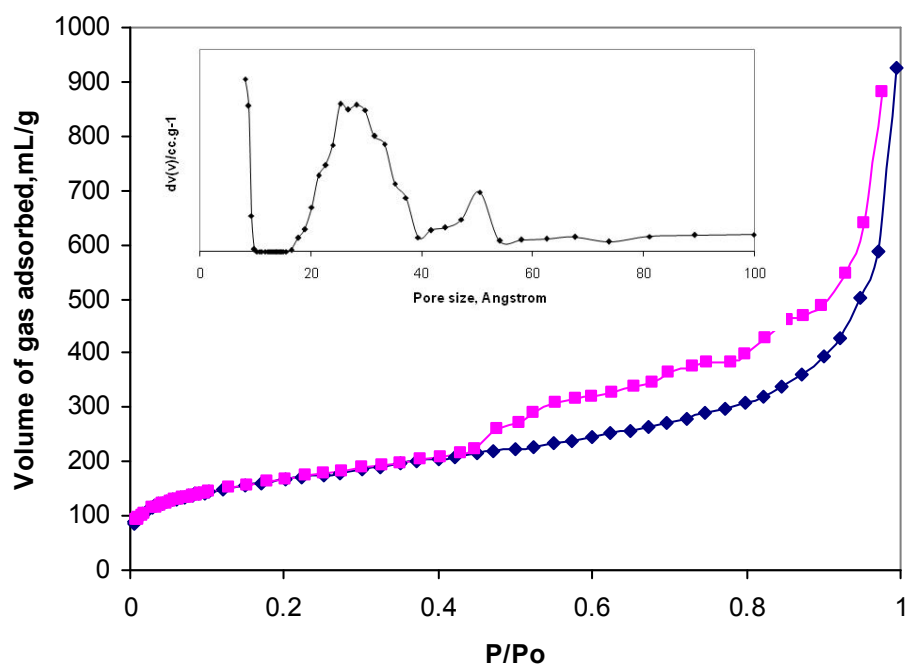

Figure S3. GPC elution profiles in (a) 50 mM phosphate buffer (pH 8) containing 10 vol % acetonitrile of PEG_{47}-b-PAA_{90} ($M_n = 1.53 \times 10^4$ and $M_w/M_n = 1.31$) and (b) THF of PEG_{47}-b-PAA_{90}-b-PSt_{80} ($M_n = 9.34 \times 10^3$ and $M_w/M_n = 1.22$).
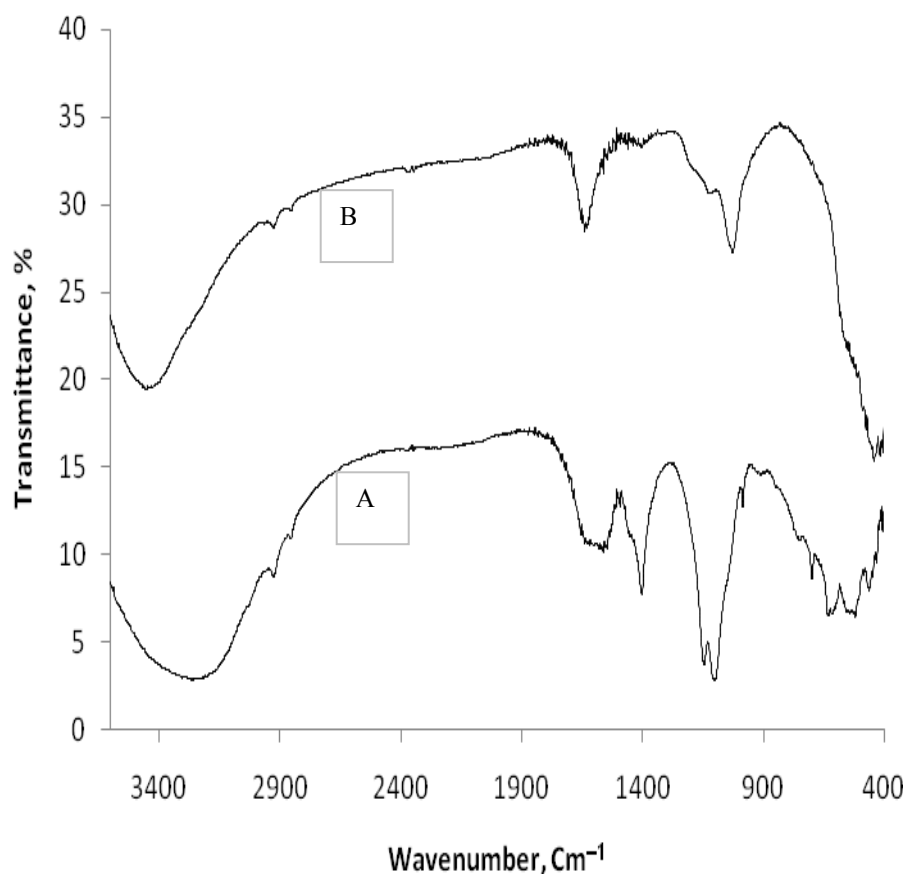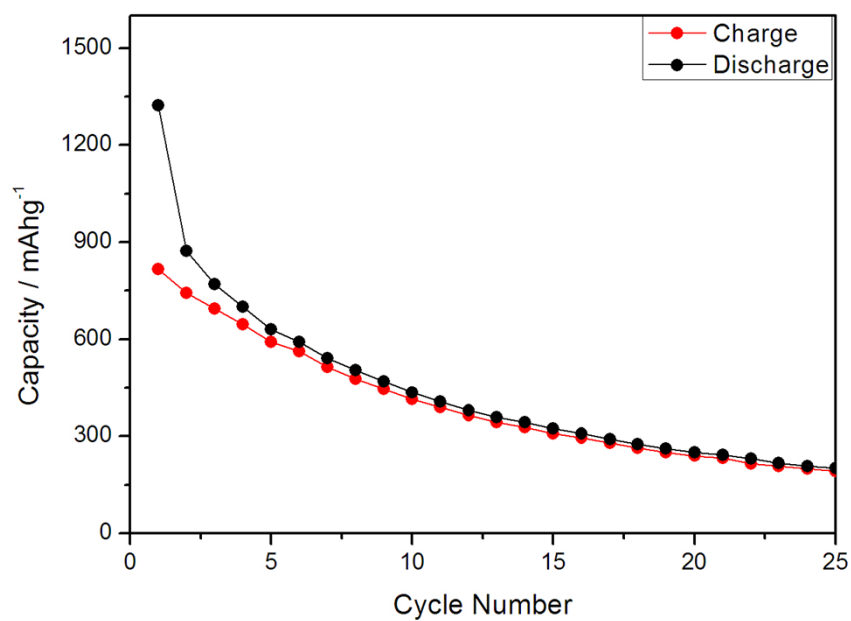
**Figure S4**. FTIR spectra of: (A) PS-PAA-PEO + Ni^{2+} composites, and (B) NiO hollow nanosphere.
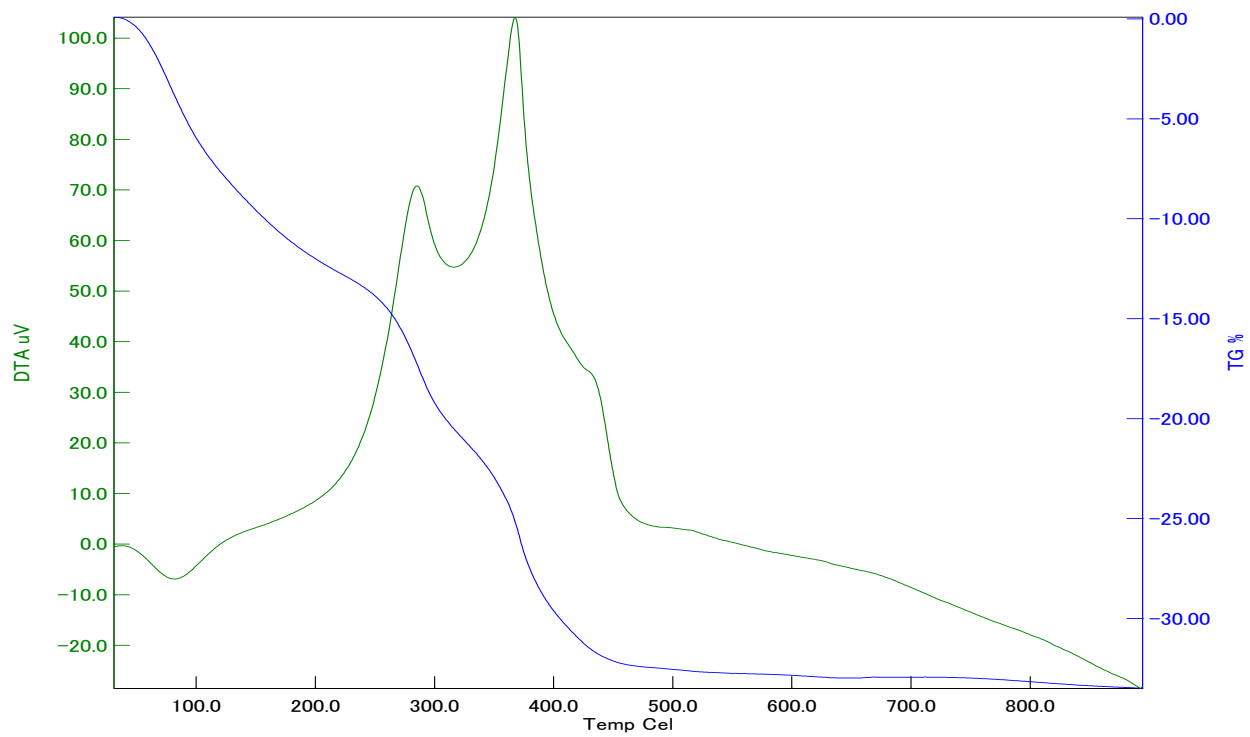
Figure S5. TG/DTA curves of PS–PAA-PEO/LaBO$_3$ composite particles.

Figure S6. Nitrogen adsorption/desorption isotherms of NiO hollow nanospheres; Inset figure represents pore-size distribution curve.
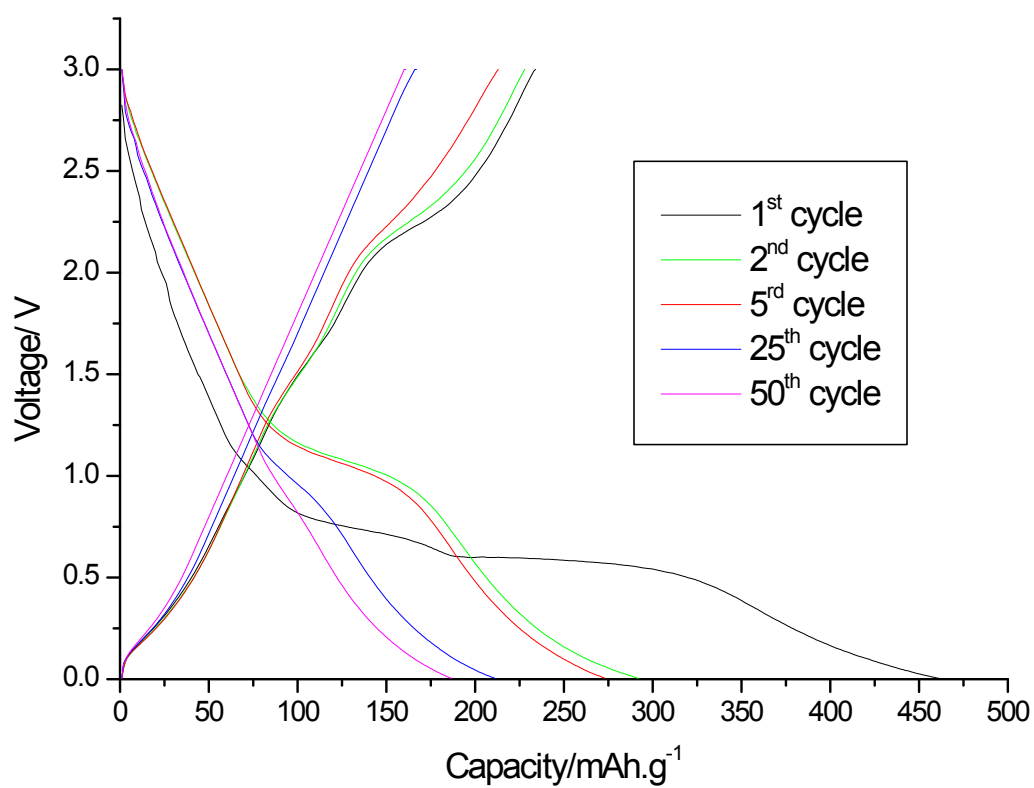
Figure S7: Cycling performance of NiO nanotubes for up to 25 cycles at a rate of 0.3 C in the voltage window of 0.005-3.0 V.
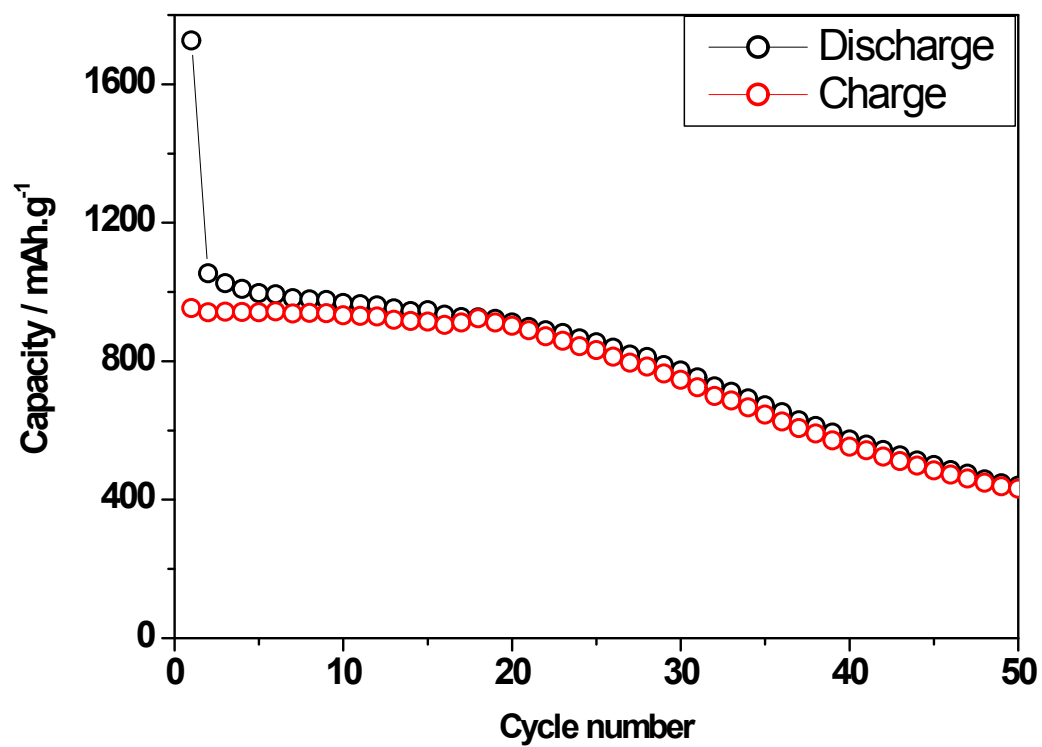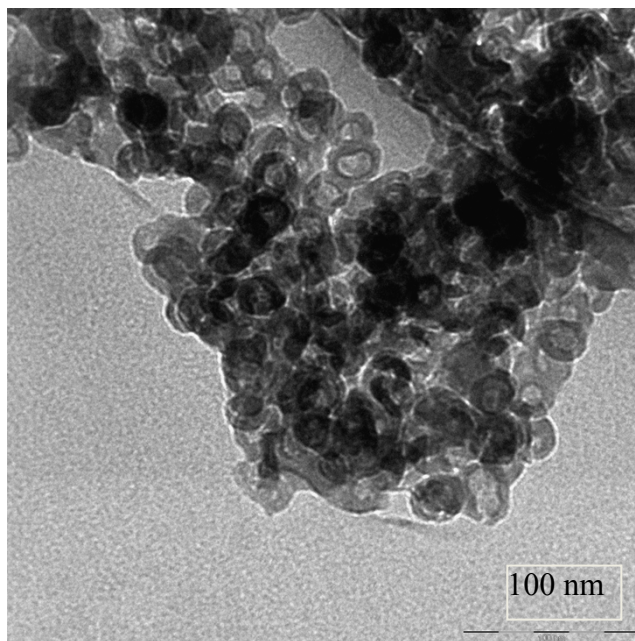
Figure S8. Rate performance of a composite containing NiO hollow particles and multiwalled carbon nanotubes (MWCNT) electrodes up to 50 cycles between 0.005 V and 3.0 V (vs. Li/Li$^+$).

Figure S9. Galvanostatic charge/discharge curves of dense NiO particle electrodes between 0.005 V and 3.0 V (vs. Li/Li$^+$).

Figure S10. TEM image of NiO hollow particles collected from the coin-cell after electrochemical characterization.