

Electronic Supplementary Information (ESI)

Hierarchical NiO Hollow Microspheres: Electrochemical and Magnetic Properties

*Hyun Gil Cha,^{‡a} Jong Hwa Sohn,^{‡a} Youngjin Park,^a Kyu Joon Lee,^b Myung Hwa Jung,^b Jae-won Lee,^c Woonsup Shin,^a Young Soo Kang^{*a}*

^a Korea Center for Artificial Photosynthesis, Department of Chemistry, Sogang University, Seoul 121-742, Republic of Korea.

^b Department of Physics, Sogang University, Seoul 121-742, Republic of Korea.

^c Department of Energy Engineering, Dankook University, Cheonan 330-714, Republic of Korea.

[‡]These authors contributed equally to the research work.

Experimental Details

a. Material Preparation.

For general synthesis, 0.02 mol of nickel chloride (NiCl_2) was dissolved in 100 ml of distilled water. Then 10 ml of nickel chloride solution was transferred to a 23 mL quantity's Teflon-lined autoclave. 10 ml of 0.4 M of ammonia water was added drop-wise into the nickel solution contained autoclave. With adding ammonia water, gel-like precipitate will appear soon. This autoclave was sealed and kept in 150 °C of convection oven for 8 h. Then it was allowed to be cooled down to room temperature. After reaction, the powder contained solution was washed with distilled water and ethanol several times with centrifugation in 5000 rpm for 5 min. The resulting pale green slurry was rinsed with DI water and several times to remove soluble impurities. The product was dried in an oven at 70 °C for 8 h to get $\text{Ni}(\text{OH})_2$. To obtain NiO the as-prepared samples was calcined at 400 °C for 4 h under air.

b. Material Characterization.

Scanning electron microscopy (SEM) images were obtained using a field-emission scanning electron microscope (Hitachi S-4300) operated at an acceleration voltage of 20 kV. A platinum/palladium alloy (in the ratio of 8 to 2) with a thickness of about 15 nm was deposited on top of the samples. Transmission electron microscope (TEM, JEM 2100F) images, selected area electron diffraction (SAED), and chemical composition were obtained from a JEOL transmission electron microscope (JEM 2100F) installed with an energy dispersive X-ray (EDX) operated at accelerating 200 keV. In these HRTEM experiments, the electron beam was incident along the direction perpendicular to the samples. The nanostructures were transferred onto TEM copper grids by dropping the solution, which was sonicated in ethanol. Powder X-ray diffraction (XRD) patterns were obtained using a Rigaku D/MAX-2500/pc diffractometer. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) data were collected from room temperature to 600 °C at a heating rate of 10 °C using a SINCO, STA-1500 analyser. Specific surface areas of the samples were obtained using N_2 adsorption-desorption at 77.3 K in Quantachrome Autosorb-1. Prior to N_2 adsorption, the samples were thermally pretreated at 100 °C under vacuum for (3×10^{-5} Torr) for 10 h. Magnetic measurements were carried out with a Quantum Design SQUID-VSM dc magnetometer. The hysteresis was recorded for powdered samples of Hierarchical $\text{Ni}(\text{OH})_2$ and NiO hollow spheres in gelatin capsule. The temperature was varied between 5 and 350 K according to a zero field cooling (ZFC) and field cooling (FC) procedure at 1 kOe, and the hysteretic loops were obtained in a magnetic field up to ± 10 kOe.

c. Electrochemical Measurements.

A coin cell (CR2016) was assembled and the electrochemical performance of the obtained samples was tested. A negative electrode was made by coating a slurry of the active material, Super P carbon black (MMM, Belgium), and poly vinylidene difluoride (PVDF) binder (Kurea, Japan) at a weight ratio of 8:1:1 onto a copper foil current collector. Li foil was used as a counter electrode. 1 M LiPF_6 in 50:50 w/w mixtures of ethylene carbonate and ethyl methyl carbonate. The coin cells were assembled in an argon-filled glovebox with the moisture and oxygen contents maintained below 1 ppm. Cyclic voltammetry (CV) measurements of the electrodes were carried out on a VSP modular 5 channel potentiostat at a scan rate of 0.1 mV/s between 0 and 3 V. Galvanostatic charge and discharge tests of the cell were carried out at constant current–constant voltage (CC-CV) mode, and the performed between 0.02 and 3.0 V at room temperature.