

Supplementary Information for;
Facile scalable synthesis of magnetite nanocrystals imbedded
in carbon matrix as superior anode materials for lithium-ion
batteries

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Experimental Section

Chemicals: The chemicals used for the preparation of the solutions were purchased at the highest grade possible. Water deionized by a Nano Pure System (Barnsted) was used. Furfuryl alcohol (99 %), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (97 %) and 20~30 nm sized magnetite nanopowder (99.9 %) were purchased from Aldrich Chemicals Corp.

Synthesis of spindle-shaped $\beta\text{-FeOOH}$ nanoparticles: Spindle-shaped $\beta\text{-FeOOH}$ nanoparticles were prepared by the hydrolysis of aqueous FeCl_3 solution. In a typical synthesis, 20 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was dissolved in 1 L of deionized water and the resulting mixture was heated at 80 °C for 12 h to obtain uniform spindle-shaped $\beta\text{-FeOOH}$ nanoparticles. The resulting nanoparticles were retrieved by centrifugation and were washed 2 times with deionized water.

Synthesis of magnetite nanocrystals imbedded in carbon matrix (Magnetite-C nanocomposite): The $\beta\text{-FeOOH}$ nanoparticles were dried in air at 90 °C for 12 h. 1 ml of furfuryl alcohol was thoroughly mixed with 3 g of the $\beta\text{-FeOOH}$ nanoparticles followed by heating at 85 °C for 12 h under a static vacuum. The composite was then heated under a nitrogen atmosphere from room temperature to 600 °C at a heating rate of 2 °Cmin⁻¹ and held at this temperature for 5 h to obtain the magnetite-C nanocomposite.

Characterization: All TEM images were obtained using a JEOL EM-2010 microscope at an acceleration voltage of 200 kV. X-ray diffraction patterns were taken by a Rigaku Dmax 2500 diffractometer system. Raman spectra were acquired with a LabRam HR spectrometer (Jobin Yvon Co., France) at an excitation wavelength of 632 nm. Elemental analysis was

performed using inductively coupled plasma atomic emission spectroscopy (ICP-AES, Shimadzu, Japan).

Electrochemical Measurements: Electrochemical studies were carried out at room temperature using two-electrode cells with lithium foil as the reference and counter electrodes. The working electrodes were prepared by mixing the magnetite-C nanocomposite, Super P, and poly(vinylidene fluoride) at a weight ratio of 70:15:15 in *N*-methyl-2-pyrrolidone solvent, and pasted on Cu foil. Polyethylene film (Celgard 2300) was used as the separator. The electrolyte solution was 1 M LiPF₆ dissolved in a 1:1 volume mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC). The coin-type half cells were assembled in an Ar-filled glove box with concentrations of moisture and oxygen of less than 1 ppm. The cells were charged and discharged at a constant current density of 100 mA g⁻¹ between 0.1 and 3.0 V (vs. Li⁺/Li) for 30 cycles with a TOSCA-3100 battery cycler (Toyo Co., Japan). Cyclic voltammetry was recorded by scanning the potential of the working electrode between 3.0 and 0.0 V (vs. Li⁺/Li) at a scan rate of 0.1 mVs⁻¹ on a Solartron multistat instrument (Model 1480, UK).

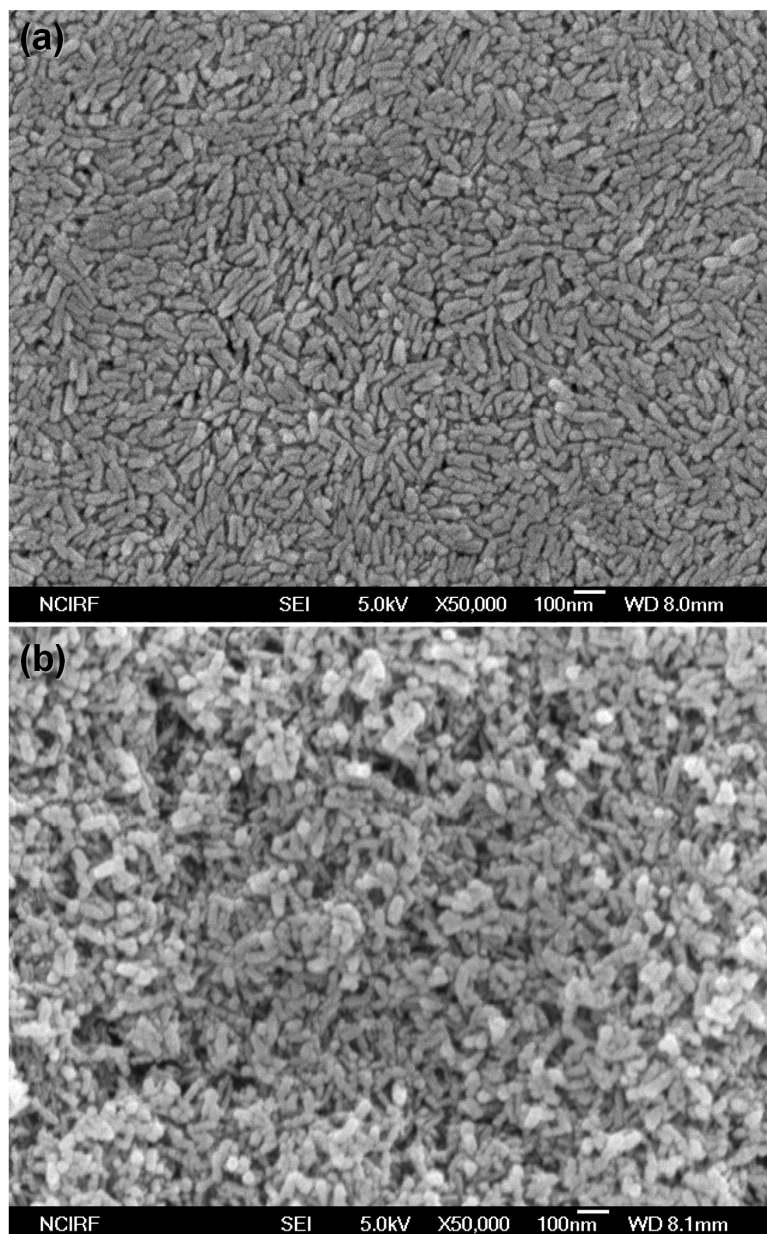


Figure S1. FE-SEM images of a) spindle-shaped β -FeOOH nanoparticles and b) the as-synthesized magnetite-C nanocomposites.

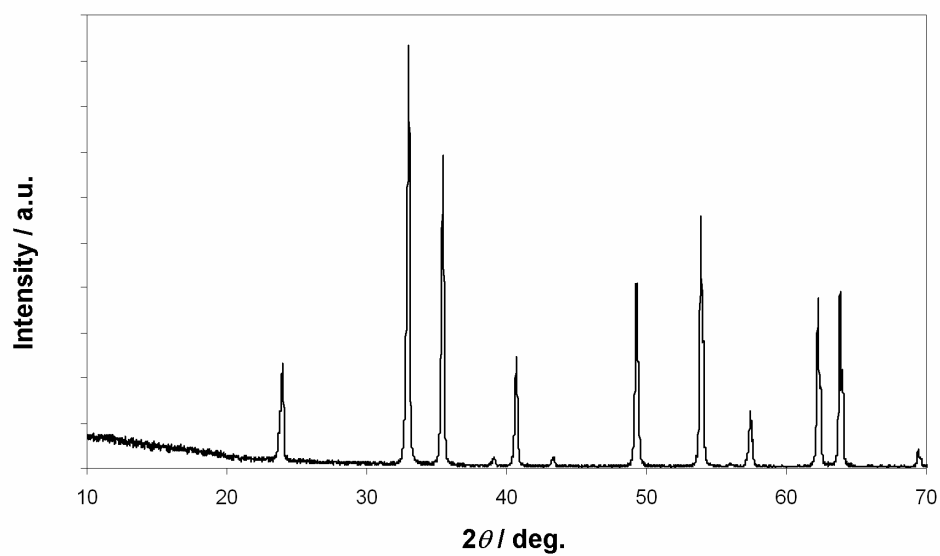
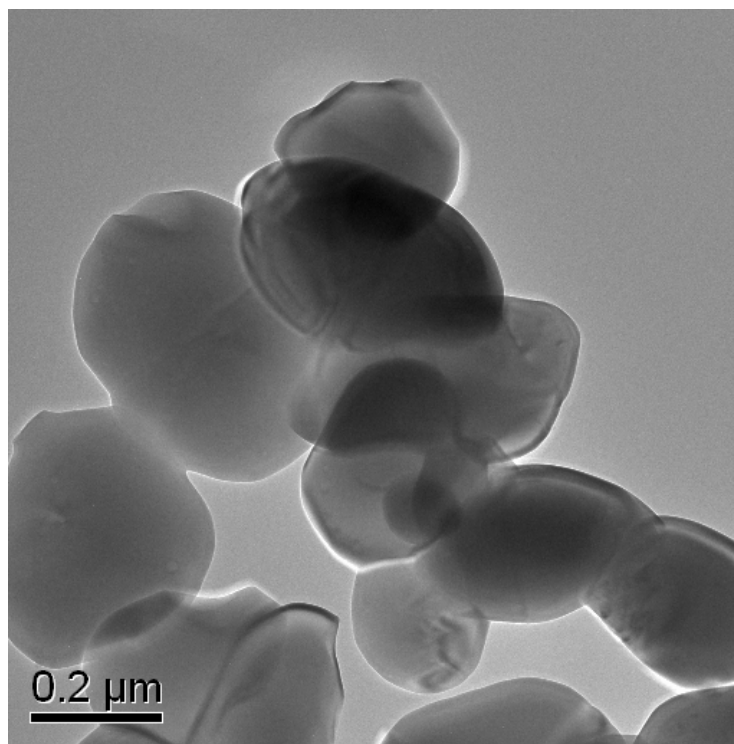


Figure S2. TEM image and XRD pattern of the hematite particles generated from heating bare $\beta\text{-FeOOH}$ nanoparticles in air.

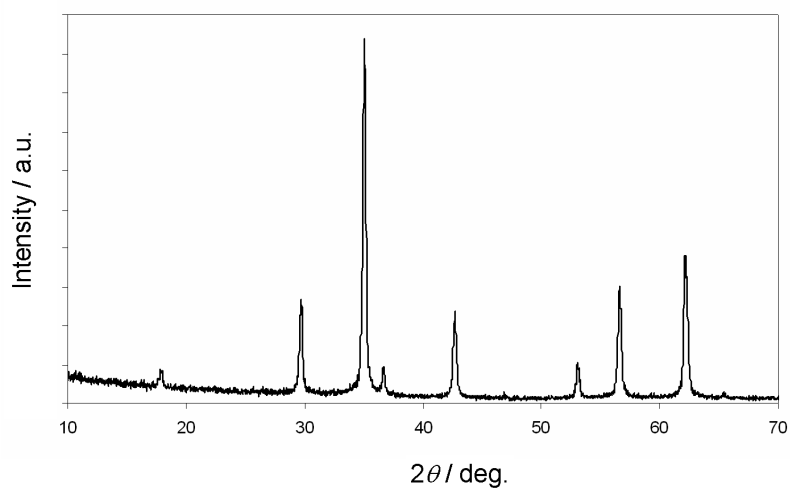
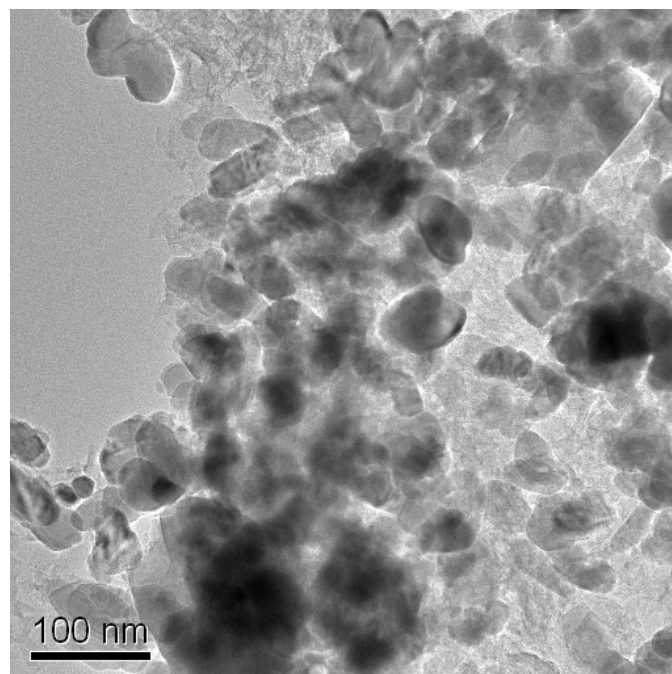


Figure S3. TEM image and XRD pattern of severely aggregated magnetite nanoparticle-carbon composites obtained from the thermal treatment of β -FeOOH-furfuryl alcohol mixture without pre-heat treatment under static vacuum.

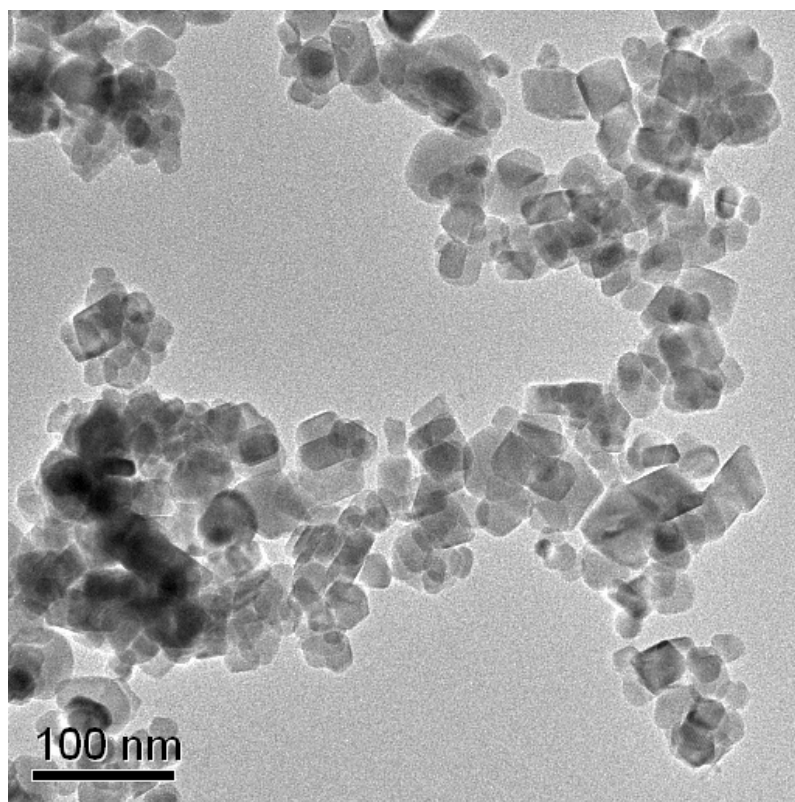


Figure S4. Typical TEM image of the commercial magnetite nanoparticles.

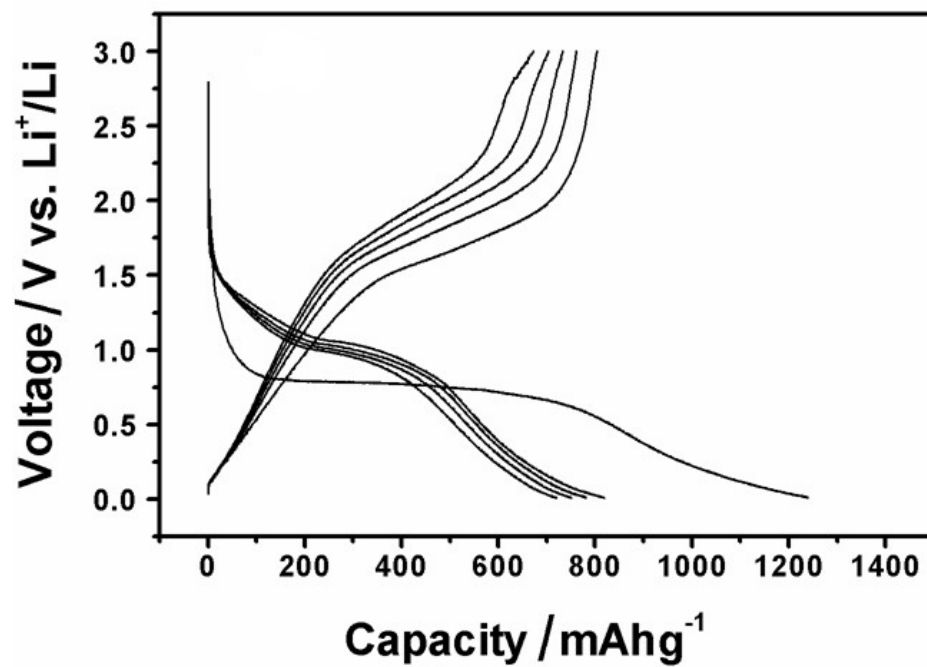


Figure S5. The discharge/charge profiles of the commercial magnetite nanoparticles.

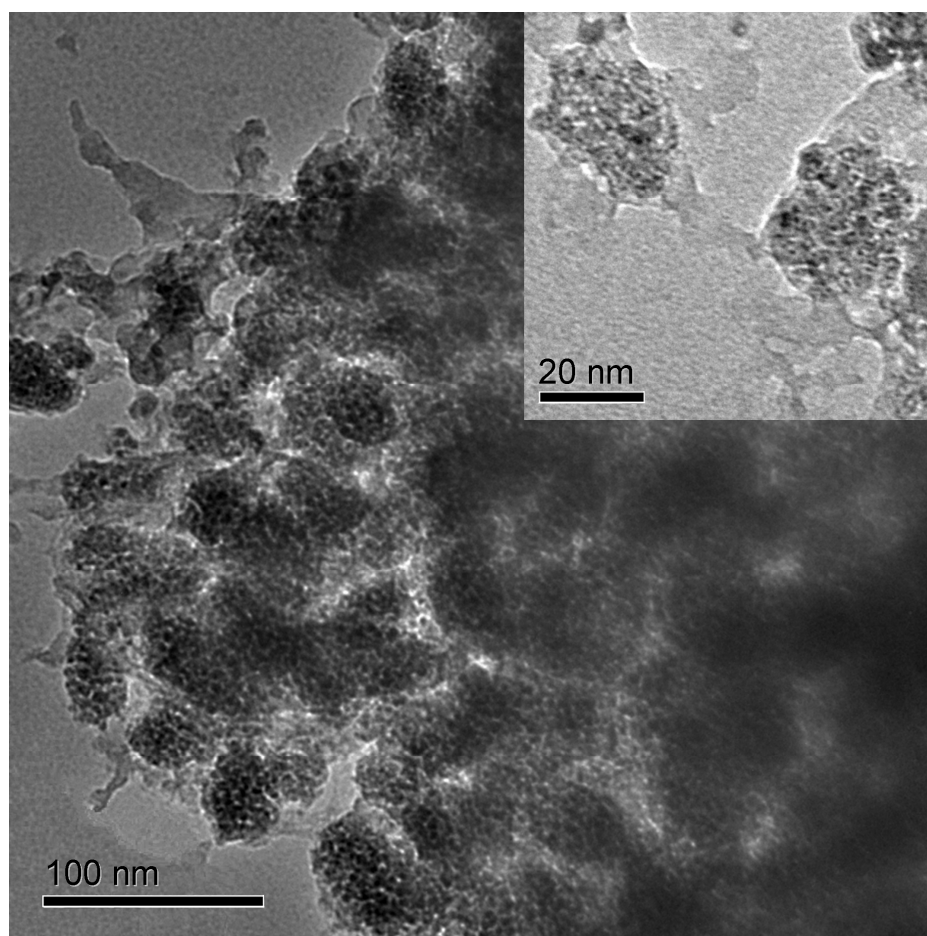


Figure S6. TEM image of the magnetite-C nanocomposites in a fully de-lithiated state after 30 cycles. The inset shows high-magnification TEM image.

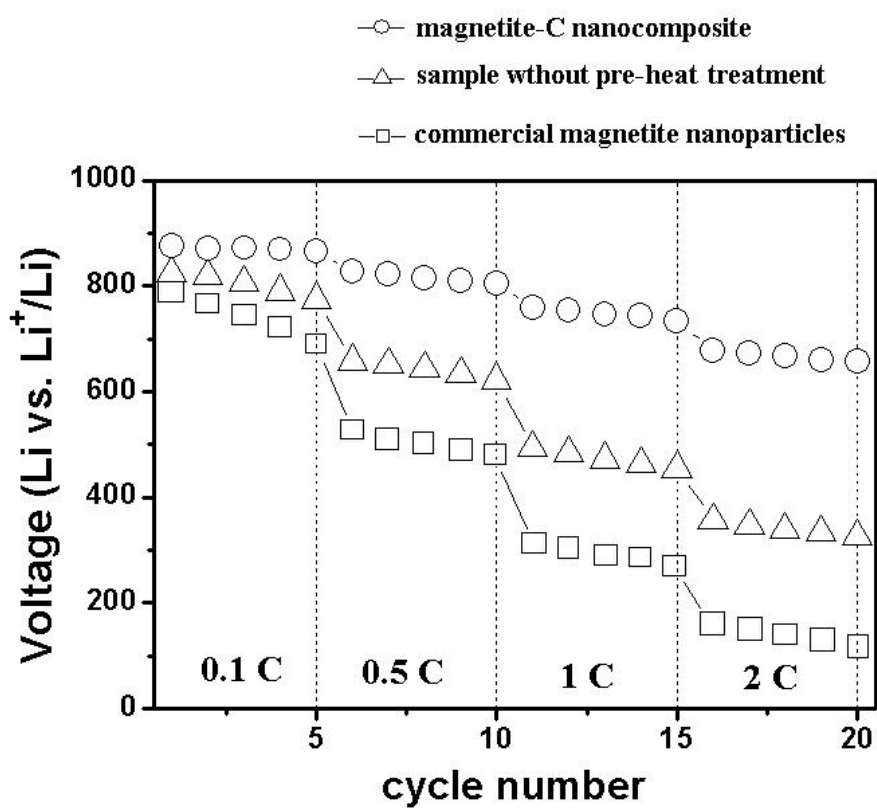


Figure S7. The plot of scan rate versus relative charge capacities for the magnetite-C composite, the sample prepared without pre-heat treatment and the commercial magnetite.



Figure S8. Photographs of (a) the β -FeOOH nanoparticles and (b) the final magnetite-C nanocomposite.