

Electronic supplementary information

Nanoporous Spherical LiFePO_4 for High Performance Cathodes†

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Experimental details

The 3D nanoporous micro-sized spherical LiFePO_4 was synthesized by spray pyrolysis followed by a brief heat treatment. The spray pyrolysis setup is shown in Figure S3. 1:1:1: 2 molar quantities of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, LiNO_3 , $\text{NH}_4\text{H}_2\text{PO}_4$ and citric acid were dissolved in a minimum amount of de-ionized water, forming a concentrated precursor solution. This solution was delivered via syringe pump to an atomizer nozzle (Sonozap Model 120K50ST, 120 kHz) to generate microdroplets, which were transported through a preheated glass tube (700 °C) by a carrier gas (5% H_2 + 95% N_2). The product collected at the end of the tube was further heat-treated at 700 °C in N_2 for 2 h to improve the conductivity of the carbon coating.

XRD patterns were collected using a Phillips X-ray diffractometer with $\text{Cu K}\alpha$ radiation. Data were collected between 15° and 75° at a scan rate of 0.01° s⁻¹. The refinement of the XRD pattern was done using RIQAS (Materials Data, Inc., Livermore, CA). SEM images were obtained using a JSM-7500F instrument (JEOL Ltd) equipped with a Thermal Scientific Inc. EDS detector used for the elemental mapping. HR-TEM images were collected with a 200 kV FEI monochromated F20 UT Tecnai microscope. TGA was performed using an SDT-Q600 analyzer (TA Instruments Inc.). The pure (carbon-free) LiFePO_4 used for TGA calibration was supplied by Hydro-Quebec Ltd (Montreal, Canada). BET measurements were carried out on a Tristar 3000 surface area & porosity analyzer (Micromeritics Instrument Corp.).

Cathodes were prepared by mixing 85 wt % LiFePO_4/C with 10 wt % carbon black and 5 wt % polytetrafluoroethylene (PTFE) binder, rolling the mixture into thin sheets, and cutting into circular electrodes of 1.26 cm² area. The electrodes had an active material loading of 4-5 mg. Coin cells assembled with these composite cathodes, metallic lithium anodes, 1 M LiPF_6 in 1:1 diethyl carbonate/ethylene carbonate electrolyte, and two pieces of Celgard 2500 polypropylene separator were cycled between 2.0 and 4.3 V.

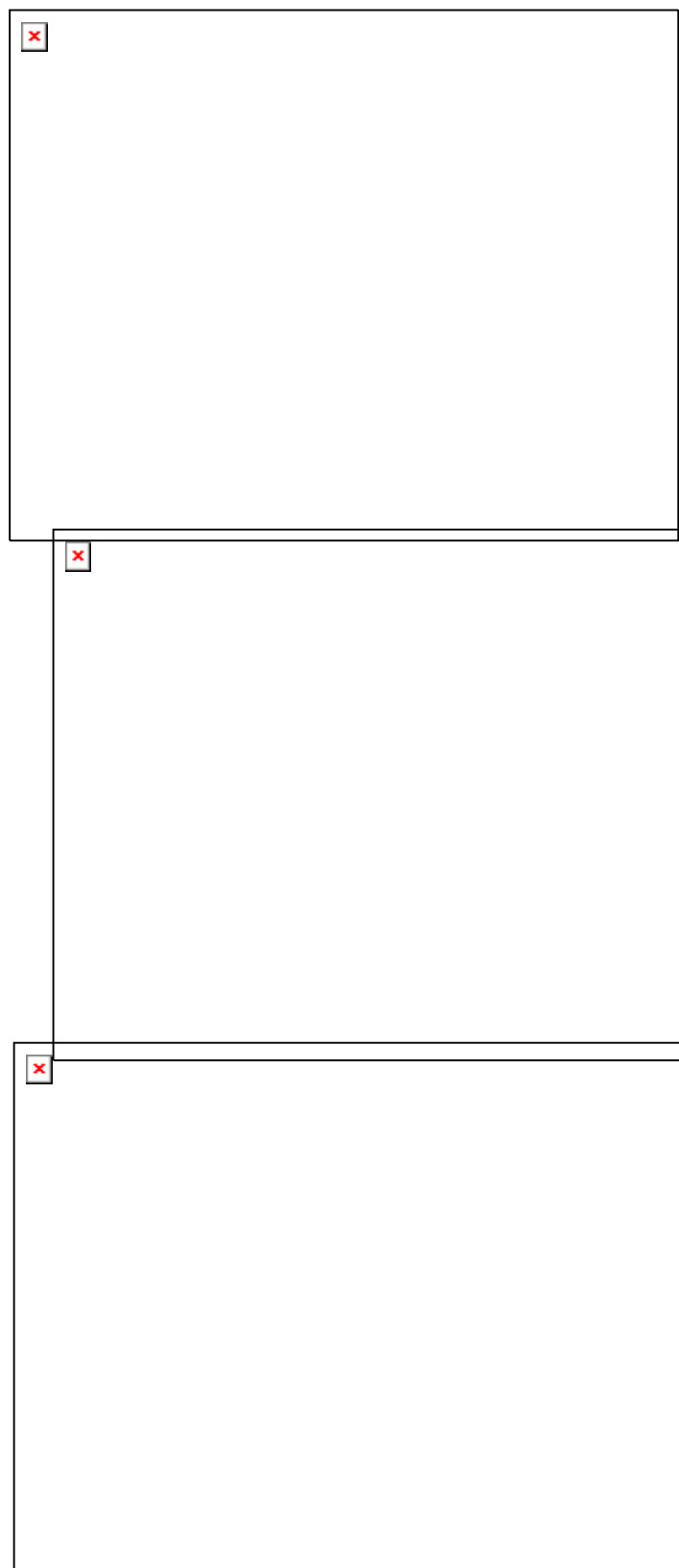


Figure S1. a) TGA curves of pure LiFePO_4 and $(\text{LiFePO}_4 + \text{C})$ mixtures; b) XRD patterns of the products of pure LiFePO_4 and $(\text{LiFePO}_4 + \text{C})$ mixtures after TGA test; c) TGA curves of pure LiFePO_4 and the 3D nanoporous spherical LiFePO_4/C .

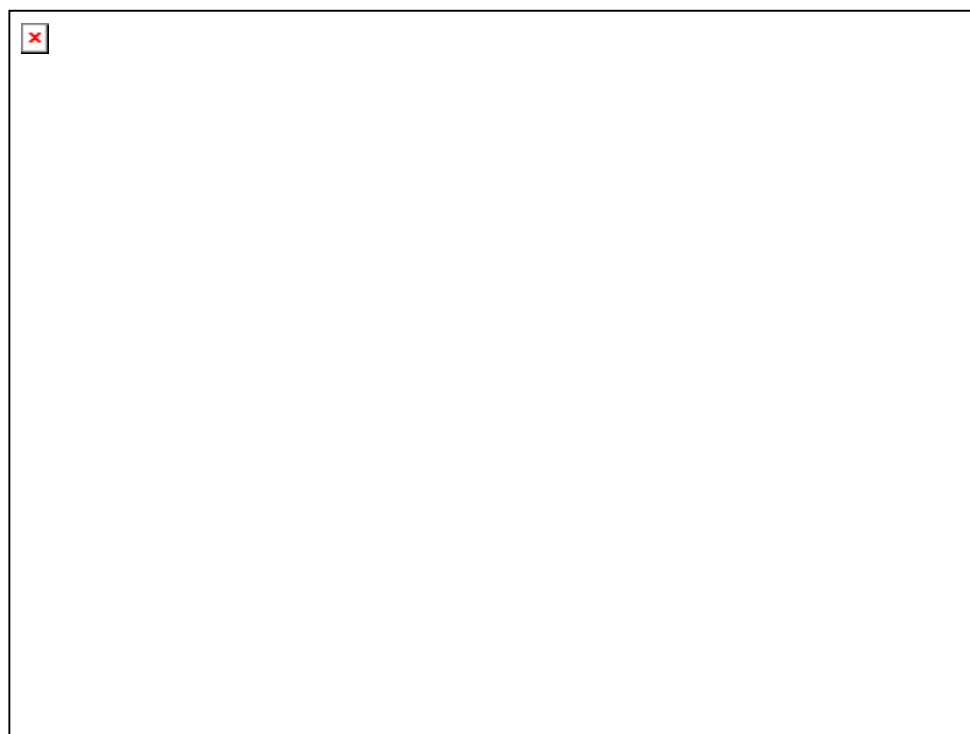


Figure S2. Nitrogen adsorption/desorption isotherms of the 3D nanoporous spherical LiFePO_4/C .

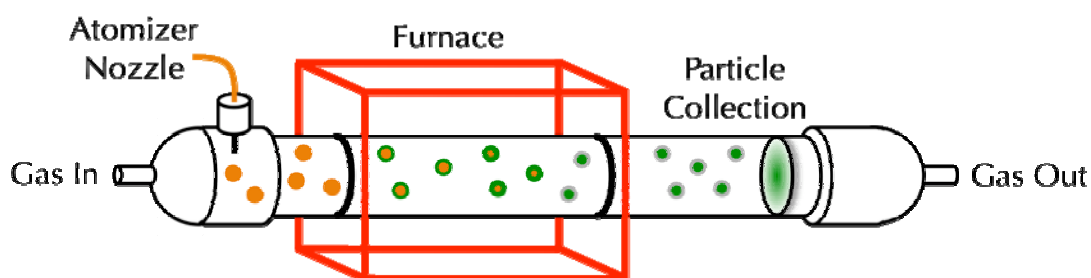


Figure S3. Schematic show of the spray-pyrolysis setup for the 3D nanoporous spherical LiFePO_4/C synthesis.

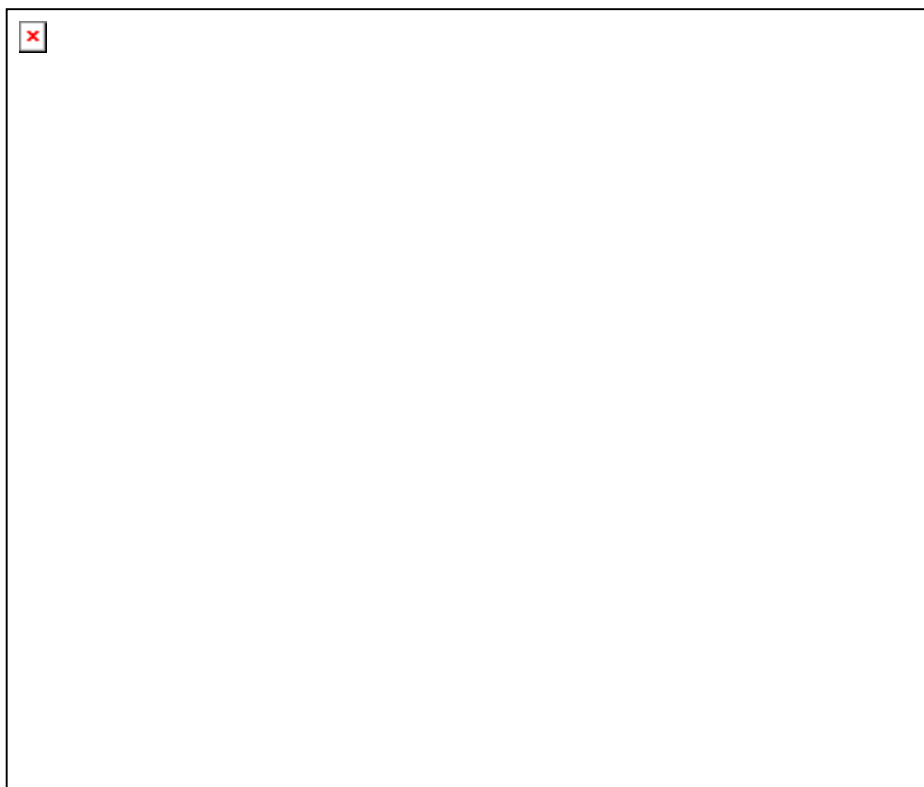


Figure S4. Cycling performance of the 3D nanoporous spherical LiFePO_4/C at 10 C.