Low temperature hydrothermal synthesis and electrochemical performances of LiFePO_4 microspheres as a cathode material for lithium-ion batteries

ZHOU Min, QIAN JiangFeng, CAO YuLiang & YANG HanXi*

Department of Chemistry, Wuhan University, Wuhan 430072, China

Received November 1, 2011; accepted December 13, 2011; published online March 31, 2012

Mesoporous LiFePO_4 microspheres were simply synthesized by a low temperature (130°C), template-free hydrothermal route using low cost LiOH , $\text{Fe}(\text{NO}_3)_3$ and $\text{NH}_4\text{H}_2\text{PO}_4$ as starting raw materials. These microspheres are composed of densely aggregated LiFePO_4 nanoparticles and filled with interconnected mesochannels, which demonstrates not only a high tap density ($\geq 1.4 \text{ g cm}^{-3}$), a high capacity of 150 mAh g^{-1} ($\sim 90\%$ of its theoretical capacity) at 0.5 C rate, but also a $\geq 80\%$ utilization of its theoretical capacity at a high rate of 1 C . In addition, the hydrothermal synthesis developed in this work is simple and cost-effective, it may provide a new route for production of the LiFePO_4 material in battery applications.

LiFePO_4 nanoparticles, mesoporous microspheres, cathodic materials, Li-ion batteries, hydrothermal synthesis

Citation: Zhou M, Qian J F, Cao Y L, et al. Low temperature hydrothermal synthesis and electrochemical performances of LiFePO_4 microspheres as a cathode material for lithium-ion batteries. *Chin Sci Bull*, 2012, 57: 4164–4169, doi: 10.1007/s11434-012-5068-4

Olivine LiFePO_4 is now intensively investigated as a promising cathodic material in rechargeable lithium ion batteries for a variety of electric storage applications such as electric vehicles (EVs) and hybrid EVs, because of its prominent advantages of high theoretical energy density as well as low material cost and environmental friendliness [1–3]. Despite these advantages, pure olivine phase of LiFePO_4 is usually difficult to realize its high electrochemical capacity at normal charge-discharge rates, due to its intrinsically low electronic conductivity and slow chemical diffusion coefficient for Li ions [3–5].

Tremendous efforts have been contributed in recent years to improve the poor electronic and ionic conduction in olivine LiFePO_4 phase by downsizing the phosphate particles [6,7], coating the particles with electronically conductive carbon or polymers [8,9] and doping the olivine lattice with supervalent cations [10–13]. Though some supervalent cations could be doped up to 3% atomic substitution in the

LiFePO_4 lattice, their contributions for electrochemical enhancement are still suspected and remains controversial [14–16]. Generally, it is now well accepted that downsizing to nanometer scale can effectively increase the electrochemical utilization of the LiFePO_4 particles, while carbon-coating can greatly help to enhance the rate capability of the material. To combine the advantages of nanosized electrode materials and conductive modification of the surface phase, various synthetic approaches, such as solid state methods [17], sol-gel routes [18,19], and hydrothermal/solvothermal synthesis [20–23], have been employed to develop nanocomposite C/LiFePO_4 particles with controlled morphologies [3–5,7]. From electrochemical point of view, microspherical LiFePO_4 particles are better suitable for lithium battery applications because of their larger packing density and better fluidity for electrode manufacture [24,25]. Recently, Xie et al. [25] and Ying et al. [26] used different co-precipitation method to obtain carbon-coated solid LiFePO_4 microspheres, however, this type of microspherical LiFePO_4 particles did not exhibit satisfactory discharge capacity and

*Corresponding author (email: hxyang@whu.edu.cn)

rate capability, possibly due to a poor utilization of the bulk phase of the solid microspheres. On the other hand, some hollow LiFePO_4 microspheres were synthesized to show enhanced electrochemical activity [27], but their volumetric densities are considerably degraded. Very recently, Yu et al. [19] reported a sol-gel/spray drying technology to obtain porous LiFePO_4 microspheres with fairly good electrochemical properties. In our recent work [28], we developed a template-free hydrothermal method to synthesize mesoporous LiFePO_4 microspheres using inexpensive Fe^{3+} salt as raw material. The LiFePO_4 microspheres of this type can not only demonstrate excellent electrochemical performances but also have a higher tap density of $\geq 1.4 \text{ g cm}^{-3}$, showing a great promise for battery applications. In this contribution, we report an improved low temperature hydrothermal synthesis and describe the synthetic chemistry for preparation of the mesoporous LiFePO_4 microspheres in detail, mainly focusing on the effects of various hydrothermal conditions on the morphology and electrochemical performances of LiFePO_4 powders.

1 Experimental

1.1 Material synthesis

The LiFePO_4 precursor was prepared by dissolving stoichiometric amounts of $\text{LiOH} \cdot \text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{NH}_4\text{H}_2\text{PO}_4$, and citric acid (molar ratio 1:1:1:1) in distilled water to form a transparent solution (70 mL, corresponding to 0.4 mol/L LiFePO_4), and then transferring the solution into a 100 mL Teflon-lined stainless steel autoclave for hydrothermal treatment. To assess the influences of hydrothermal conditions on the morphology and size of the LiFePO_4 precursor, the hydrothermal treatment were carried out at different temperatures or with different reaction times respectively. When the hydrothermal reactions finished, the autoclave was cooled down naturally to room temperature and the reaction solution was evaporated at 80°C under continuously stirring to obtain a light green spherical precursor. The precursor was calcined at 650°C for 10 h under argon containing 5% H_2 to obtain pure LiFePO_4 microspheres. To coat with carbon, the precursor was impregnated in a sucrose solution (sucrose: LiFePO_4 =0.25:1 by wt.) and then dried and calcined in the same condition as mentioned above.

1.2 Structural characterization

The crystalline structures of the hydrothermal precipitates and products were characterized by powder X-ray diffraction (XRD) using a Shimadzu XRD-6000 diffractometer equipped with $\text{CuK}\alpha$ radiation. The XRD spectra were collected in a range of 2θ values from 10° to 70°C at a scanning rate of $1^\circ/\text{min}$ and a step size of 0.02°C . The morphologies

of the as-synthesized samples were observed by scanning electron microscopy (SEM, Sirion 2000, FEI) and the nanostructures of the samples were examined by transmission electron microscopy (TEM, JEM-2010, FEF). The samples for the TEM analysis were prepared by dispersing the sample powders in ethanol and releasing a few drops of the dispersed solution on a carbon film supported on a copper grid. Nitrogen adsorption-desorption isotherms for the surface-area and pore analysis of the samples were measured with an ASAP 2020 (Micromeritics Instruments). Thermogravimetric measurement (TG) was conducted on a TGA Q500 thermogravimetric analyzer (TA Instruments, USA) in oxygen at a scan rate of $10^\circ\text{C}/\text{min}$ from room temperature to 700°C .

1.3 Electrochemical measurements

Electrochemical performance characterization of the as-prepared LiFePO_4 materials measurements were carried out using a 2016-type coin cell. The working electrodes were made by pressing a 0.8 cm^2 thin film (containing 80 wt% LiFePO_4/C composite, 12 wt% acetylene black, and 8 wt% polytetrafluoroethylene) onto a Al mesh. The counter electrode was a disk of lithium metal foil and the electrolyte was a 1 mol L^{-1} LiPF_6 dissolved in a mixture of ethylene carbonate, dimethyl carbonate and ethylmethyl carbonate (1:1:1 by vol., Shinnest Battery Materials Co. Ltd., China). The charge-discharge capacities of the LiFePO_4/C composites were calculated on the basis of net weight of LiFePO_4 , exclusive of the carbon content. The test cells were assembled in an argon-filled glove box and the galvanostatic charging-discharging experiments were conducted by a BTS-55 Neware battery testing system at various rates ($1 \text{ C} = 150 \text{ mA g}^{-1} \text{ LiFePO}_4$) in the voltage range 2.0–4.3 V at room temperature.

2 Results and discussion

2.1 Growth of LiFePO_4 microspheres in hydrothermal environment

A key factor in determining the morphology and size of a precipitate in hydrothermal synthesis is to control the reaction temperature. Usually, higher temperature can promote the precipitation and grain growth of the solid phase in a hydrothermal environment, but increasing temperature can also bring about a number of difficulties, such as energy consumption and safety control, from the viewpoint of industrial production. Previous hydrothermal synthesis of morphologically controlled LiFePO_4 particles was almost carried out at the temperatures higher than 180°C [20,23]. To search for a low temperature synthesis, we examined the grain growth and morphological evolution of the LiFePO_4 precursors at a wider temperature range from 100°C to 200°C .

Figure 1 shows the SEM images of the LiFePO_4 precursors obtained at various hydrothermal temperatures at a fixed reaction time of 6 h.

It can be clearly visualized from the images in Figure 1 that when the hydrothermal temperature was set at $\leq 100^\circ\text{C}$, the LiFePO_4 precursors could only precipitate into irregular sub-micrometered particles but not in spherical morphology. At increased temperature to 120°C , the precipitates tended to agglomerate and produce fragmented microspheres. As long as the reaction temperature rose to $\geq 130^\circ\text{C}$, the LiFePO_4 precursors appeared entirely as well-shaped microspheres and the size of the microspherical precursors decreased slightly with increasing temperature from 130 to 200°C . Very differently, the precursor microspheres exhibited a rather smooth surface as prepared at the temperature of 130 – 160°C , whereas the microspherical particles obtained at 180 – 200°C showed a rough surface with obvious porous structure. Since the spheroidizing reaction of the LiFePO_4 precursors can take place at a fairly low temperature of 130°C , it is our interest to develop a synthetic route for LiFePO_4 particles at this low hydrothermal temperature.

Figure 2 shows the growth and morphological changes of the hydrothermal precipitate at different durations in the hydrothermal reaction at 130°C . As is shown in the SEM images of Figure 2, the precursors started to precipitate into irregular nanoparticles at the first hour of the hydrothermal treatment and became spherically shaped when they were hydrothermally treated for 2 h. With increasing the reaction time, the precursor particles gradually grew and their surfaces turned into a rugged and porous structure under the

Figure 1 SEM images of the LiFePO_4 precursors after hydrothermal reaction of 6 h at temperature of 100°C (a), 120°C (b), 130°C (c), 160°C (d), 180°C (e) and 200°C (f), respectively.

Figure 2 SEM images of the precursor produced hydrothermally at 130°C for different time. (a) 1 h; (b) 2 h; (c) 6 h; (d) 12 h.

hydrothermal reaction for 6 h.

This dramatic change in the morphology and size of the LiFePO_4 precursors with the hydrothermal conditions could be accounted for by a dissolution-precipitation mechanism [28,29]. In this mechanism, the hydrothermal reaction mainly proceeded in three steps: hydrolytic nucleation, crystal growth and morphological evolution. At the initial stage of the hydrothermal reaction, the metallic Fe^{3+} and Li^+ cations hydrolyzed to create their saturated concentrations and then precipitated to form the irregular nanosized aggregates, which consisted of iron phosphate hydroxides and lithium phosphate, as detected from a XRD analysis. As the reaction progressed, the aggregated nanoparticles grew up at the increased temperature and then agglomerated into microspheres at elevated internal pressure, due to the thermodynamic requirement for minimizing the surface free energy of the aggregates. When the reaction lasted for a long time, the freshly agglomerated microspheres must undergo a structural reconstruction through a dissolution-deposition process under a cooperative action of high temperature and pressure, meanwhile smaller isolated crystallites on the edged surfaces dissolved into the solution and some neighboring crystallites developed to form larger particles, finally leading to porous microspheres. In our experiments, the porous microspherical LiFePO_4 precursors can be prepared as long as the hydrothermal temperature maintained at $\geq 130^\circ\text{C}$, but the hydrothermal reaction at lower temperatures needed longer time to create the interconnected pore chains in the spherical particles.

In addition, the reactant concentrations can also considerably affect the size and morphology of the precursor particles. Fixing the hydrothermal temperature at 130°C , we compared the morphologies of the precursor particles deposited from different solutions with the reactant concentrations equally varying from 0.1 to 0.8 mol L^{-1} and found that a similar porous microspherical morphology could be observed so far as the molar concentrations of the Fe^{3+} , Li^+ and PO_4^{3-} ions were all higher than 0.4 mol L^{-1} , and other-

wise the hydrothermal products appeared as irregular nanoparticles. This concentration effect may arise from the reason that the reactants with very low concentrations could not produce adequate amount of the precipitates and sufficiently high internal pressure that were necessarily required for creating spherical particles.

2.2 Structure characterizations

In order to achieve better electrochemical performances, we impregnated the porous microspheres in a sucrose solution and then calcined the precursors to obtain the carbon-coated LiFePO_4 microspheres (denoted as C/LiFePO₄ or C/LFP) as described in experimental section. The C/LFP powders obtained from the precursors hydrothermally treated at 130 and 180°C are marked correspondingly as C/LFP-130 and C/LFP-180, respectively. Figure 3 compares the XRD patterns of the C/LiFePO₄ powders synthesized hydrothermally at 130°C (C/LFP-130) and at 180°C (C/LFP-180). All the peaks in the XRD patterns could be indexed to an orthorhombic space group, *Pnma* (JCPDF card no: 40-1499), suggesting a pure olivine phase of the as-synthesized samples. By using Debye-Scherrer's formula, the mean size of the LiFePO_4 crystallites in the C/LFP-130 and C/LFP-180 samples was calculated to be ~80 nm, which is consistent with the SEM observations.

TEM can provide an insight into the microstructure details of the C/LiFePO₄ spheres. As shown in Figure 4(a), the C/LiFePO₄ microspheres are actually composed of nanoparticles embedded in the carbon matrix and each nanoparticle has a crystal size of about 60–80 nm, fairly agreeing with the value calculated from the XRD data. From the HRTEM image of a single crystallite (Figure 4(b)), it can be seen clearly that the carbon layer is intimately coated on the surface of the LiFePO_4 crystallites showing as an irregular 2 nm thick strip on the crystallite surface and in the interior

Figure 3 X-ray diffraction (XRD) patterns of the C/LiFePO₄ samples obtained from the hydrothermally LiFePO_4 precursors synthesized at 130 and 180°C as labeled.

Figure 4 (a) Transmission electron microscopy (TEM) image of the selected surface of a C/LiFePO₄ microparticle; (b) the HRTEM image of a LiFePO_4 crystallite at 180°C for 6 h.

region, the magnified HRTEM image shows a regular lattice fingerprint of the (021) plane ($d = 0.348$ nm) of orthorhombic LiFePO_4 , indicating a perfectly crystallized LiFePO_4 phase in the cores of each particles.

Based on the measurement of the carbon amount released from the samples during the temperature scan in oxygen atmosphere, the carbon contents in the C/LFP-130 and C/LFP-180 samples were calculated to be ~14.24% and ~5.79%, respectively, suggesting the different mesoporous structures in the samples obtained at the different hydrothermal temperatures. To evaluate the porous structure and attainable surface area, we measured the adsorption and desorption isotherms of the LFP, C/LFP-130 and C/LFP-180 samples. In general, the adsorption isotherm for pure LiFePO_4 particles appears as a type III curve, suggesting that the pores are distributed in the macroporous range. In contrast, the C/LFP-130 and C/LFP-180 samples show an IV type adsorption isotherm with a large H_2 -type hysteresis loop and clear plateaus at relative pressures of 0.4–0.9, which are characteristic of molecular adsorption in mesopores. Detailed analysis demonstrates that the mesopores are predominately distributed in the diameter region of 50–100 nm. Calculated from nitrogen adsorption isotherms, the BET surface areas of the LFP, C/LFP-130 and C/LFP-180 powders are 8.4, 33.4 and 40.0 $\text{m}^2 \text{g}^{-1}$, respectively. Apparently, the increased surface area of the carbon-coated samples arose from the surface layer of pyrolyzed carbon. Nevertheless, such a mesoporous structure favors the electrolyte penetrating into the interior voids of the C/LiFePO₄ microspheres, so as to achieve a higher transport and a better utilization of the material.

2.3 Electrochemical performances

Figure 5 shows the charge-discharge performances of the C/LFP-130 and C/LFP-180 samples at various discharge rates. At a moderate rate of ~0.3 C (50 mA g^{-1}), both the C/LFP-130 and C/LFP-180 electrode can deliver a discharge capacity of 155 and 150 mAh g^{-1} , respectively, corresponding to a nearly full utilization of LiFePO_4 (theoretical

Figure 5 Charge-discharge curves of the C/LFP-130 (a), and the C/LFP-180 electrodes at different rates (b). (c) Cycling performances of the C/LFP-130 and the C/LFP-180 electrodes.

capacity: $\sim 160 \text{ mAh g}^{-1}$). At a quite high rate of 1 C, the C/LFP-130 electrode can still give a high capacity of $\sim 135 \text{ mAh g}^{-1}$, while the C/LFP-180 electrode shows a higher capacity of $\sim 140 \text{ mAh g}^{-1}$, which is about 87% of their theoretical capacity. These discharge capacities are almost the same as those reported from the highly active LiFePO_4 nanoparticles but much higher than those commonly reported LiFePO_4 nanopowders [21,22]. Figure 5(c) shows the cycling performances of the C/LFP-130 and C/LFP-180 samples. At the charge and discharge rate of $\leq 0.5 \text{ C}$, both the electrodes exhibit a similar reversible capacity. But, with the cycling current increased to 1 C, the C/LFP-180 electrode shows higher rate capability than the C/LFP-130 elec-

trode. This difference in the rate capability is most likely due to an increased surface area and higher porous volume of the microspheres hydrothermally prepared at 180°C . Nevertheless, all the LiFePO_4 microspheres thus prepared show a very stable charge-discharge capacity with almost no discernible capacity decay during cycling from 0.3 to 1.0 C rate, implying good capacity retention as cathode-active materials.

A striking feature of the as-prepared LiFePO_4 microspheres is its high tap density of $\sim 1.4 \text{ g cm}^{-3}$, which is $\sim 40\%$ higher than currently reported nano- LiFePO_4 materials ($\leq 1.0 \text{ g cm}^{-3}$). Though some solid LiFePO_4 micro-particles were reported to have a higher tap density ($\geq 1.6 \text{ g cm}^{-3}$) [25,30], their electrochemical utilization and rate capability are remarkably lower than LiFePO_4 nanoparticles. In contrast, the LiFePO_4 microspheres prepared in this work are composed of densely packed nanoparticles, forming abundant electrochemical interfaces and three-dimensional mesochannels, which promote not only the interfacial intercalation reaction but also the electrolyte transport in the interior of the mesoporous microspheres, so as to reach a good compromise between the volumetric and gravimetric electric storage densities.

3 Conclusions

In this work, mesoporous LiFePO_4/C microspheres were prepared by a template-free hydrothermal route at low temperature of 130°C using low cost LiOH , $\text{Fe}(\text{NO}_3)_3$ and $\text{NH}_4\text{H}_2\text{PO}_4$ as starting iron source and a subsequent carbon thermal reduction process. These microspheres are composed of densely packed LiFePO_4 nanoparticles and filled with interconnected mesochannels, which allow better penetration of electrolyte and can therefore provide abundant electrochemically effective surfaces for lithium intercalation reaction. In addition, such a microspherical structure enables the aggregated LiFePO_4 nanoparticles to reach a quite high volumetric energy density along with a high efficient utilization of the material. Since the hydrothermal synthesis is facile and cost-effective, it may provide a new route for production of C/ LiFePO_4 in battery applications.

This work was supported by the National Basic Research Program of China (2009CB220103).

- 1 Padhi A K, Nanjundaswamy K S, Goodenough J B. Phospho-olivines as positive-electrode materials for rechargeable lithium batteries. *J Electrochem Soc*, 1997, 144: 1188–1194
- 2 Wang Y, Cao G. Developments in nanostructured cathode materials for high-performance lithium-ion batteries. *Adv Mater*, 2008, 20: 2251–2269
- 3 Yuan L, Wang Z, Zhang W, et al. Development and challenges of LiFePO_4 cathode material for lithium-ion batteries. *Energy Environ Sci*, 2010, 4: 269–284
- 4 Kang B, Ceder G. Battery materials for ultrafast charging and dis-

- charging. *Nature*, 2009, 458: 190–193
- 5 Wang Y, He P, Zhou H. Olivine LiFePO₄: Development and future. *Energy Environ Sci*, 2011, 4: 815–817
 - 6 Gaberscek M, Dominko R, Jamnik J. Is small particle size more important than carbon coating? An example study on LiFePO₄ cathodes. *Electrochem Commun*, 2007, 9: 2778–2783
 - 7 Wang Y, Hosono E, Wang K, et al. The design of a LiFePO₄/carbon nanocomposite with a core-shell structure and its synthesis by an *in situ* polymerization restriction method. *Angew Chem (Int. ed)*, 2008, 47: 7461–7465
 - 8 Ravet N, Goodenough J B, Besner S. In the 196th Meeting of the Electrochemical Society, Honolulu, 1999
 - 9 Huang Y H, Goodenough J B. High-rate LiFePO₄ lithium rechargeable battery promoted by electrochemically active polymers. *Chem Mater*, 2008, 20: 7237–7241
 - 10 Chung S Y, Bloking J T, Chiang Y M. Electronically conductive phospho-olivines as lithium storage electrodes. *Nat Mater*, 2002, 1: 123–128
 - 11 Ellis B, Kan W H, Makahnouk W R M, et al. Synthesis of nanocrystals and morphology control of hydrothermally prepared LiFePO₄. *J Mater Chem*, 2007, 17: 3248–3254
 - 12 Subramanya H, Ellis B, Coombs N, et al. Nano-network electronic conduction in iron and nickel olivine phosphates. *Nat Mater*, 2004, 3: 147–152
 - 13 Zeng L J, Gong Q, Liao X Z, et al. Enhanced low-temperature performance of slight Mn-substituted LiFePO₄/C cathode for lithium ion batteries. *Chin Sci Bull*, 2010, 55: 1262–1266
 - 14 Chung S Y, Choi S Y, Yamamoto T, et al. Orientation-dependent arrangement of antisite defects in lithium iron (II) phosphate crystals. *Angew Chem*, 2009, 48: 543–546
 - 15 Hong J, Wang C S, Chen X, et al. Vanadium modified LiFePO₄ cathode for Li-ion batteries. *Electrochem Solid-State Lett*, 2009, 12: A33–A38
 - 16 Islam M S, Driscoll D J, Fishe C A J, et al. Atomic-scale investigation of defects, dopants, and lithium transport in the LiFePO₄ olivine-type battery material. *Chem Mater*, 2005, 17: 5085–5092
 - 17 Hu Y S, Guo Y G, Dominko R, et al. Improved electrode performance of porous LiFePO₄ using RuO₂ as an oxidic nanoscale interconnect. *Adv Mater*, 2007, 19: 1963–1966
 - 18 Dominko R, Bele M, Gaberscek M. Porous olivine composites synthesized by sol-gel technique. *J Power Sources*, 2006, 153: 274–280
 - 19 Yu F, Zhang J J, Yang Y F, et al. Up-scalable synthesis, structure and charge storage properties of porous microspheres of LiFePO₄/C nanocomposites. *J Mater Chem*, 2009, 19: 9121–9125
 - 20 Chen J, Whittingham M S. Hydrothermal synthesis of lithium iron phosphate. *Electrochem Commun*, 2006, 8: 855–858
 - 21 Dokko K, Koizumi S, Sharaishi K, et al. Electrochemical properties of LiFePO₄ prepared by hydrothermal route. *J Power Sources*, 2007, 165: 656–659
 - 22 Recham N, Dupont L, Courty M. Ionothermal synthesis of tailor-made LiFePO₄ powders for Li-ion battery applications. *Chem Mater*, 2009, 21: 1096–1107
 - 23 Saravanan K, Balaya P, Reddy M V, et al. Morphology controlled synthesis of LiFePO₄/C nanoplates for Li-ion batteries. *Energy Environ Sci*, 2010, 3: 457–464
 - 24 He X, Li J, Cai Y, et al. Preparation of co-doped spherical spinel LiMn₂O₄ cathode materials for Li-ion batteries. *J Power Sources*, 2005, 150: 216–222
 - 25 Xie H M, Wang R S, Ying J R. Optimized LiFePO₄-polyacene cathode material for lithium-ion batteries. *Adv Mater*, 2006, 18: 2609–2613
 - 26 Ying J, Lei M, Jiang C. Preparation and characterization of high-density spherical Li_{0.97}Cr_{0.01}FePO₄/C cathode material for lithium ion batteries. *J Power Sources*, 2006, 158: 543–549
 - 27 Yang M R, Teng T H, Wu S H. LiFePO₄/carbon cathode materials prepared by ultrasonic spray pyrolysis. *J Power Sources*, 2006, 159: 307–311
 - 28 Qian J F, Zhou M, Cao Y L, et al. Template-free hydrothermal synthesis of nanoembossed mesoporous LiFePO₄ microspheres for high-performance lithium-ion batteries. *J Phys Chem C*, 2010, 114: 3477–3482
 - 29 Qin X, Wang X, Xiang H J. Mechanism for hydrothermal synthesis of LiFePO₄ platelets as cathode material for lithium-ion batteries. *Phys Chem C*, 2010, 114: 16806–16812
 - 30 Dominko R, Bele M, Goupil J M. Wired porous cathode materials: A novel concept for synthesis of LiFePO₄. *Chem Mater*, 2007, 19: 2960–2969

Open Access This article is distributed under the terms of the Creative Commons Attribution License which permits any use, distribution, and reproduction in any medium, provided the original author(s) and source are credited.