Electronic Supplementary Material (ESI) for ChemComm.

Supporting information

Experimental Section

Material and electrode

Material preparation:

The $FePO_4$ material was prepared by the liquid phase precipitation method with a scalable and simple procedure, and can achieve small size and uniform distribution particles. And, a stoichiometric Fe/P ratio of 1 is used. The $Fe(NO_3)_3$ was chose as iron sources. The raw material of $(NH_3)_2HPO_4$ was used as phosphrous sources. In addition, the H_3PO_4 was added into the solution to adjust the pH=2.0. The iron sources solution was controlled strictly to add into the phosphrous sources solution by a peristaltic pump with the quite low flow rate at the 70 °C. And the pale-yellow precipitate was obtained in solution by the repeat filter. The synthesized-solid was disposed in the pipe furnace of Ar gas atmosphere at 380 °C. After a 60-min and then cooling to room temperature, the $FePO_4$ was washed with deionized water and ethanol several times. At last, the product was dried at air at 100 for 12h. The $FePO_4$ material was as an anode to use in the S-DIBs. For the cathode material, layered-graphite (Alfa Aesar, 325 Mesh, 99.8%) was used as the intercalation host of the PF_6^- anion.

Electrode preparation: the graphite (anode) or $FePO_4$, polyvinylidene fluoride (PVDF) and conductive agent (Super-P) were mixed in a weight ratio of 8:1:1 in N-methyl pyrrolidinone (NMP) solvent. The mixture was mechanically mixed for 12 hrs. The resultant slurry was coated onto Al-foil (for cathode/anode electrode) by an automatic coating machine. The prepared electrode was dried at 110 °C under vacuum for 6 hours to evaporate the water.

Electrochemical tests

For the above electrodes, the thickness was strictly controlled by the thickness control mold. The proportion of loading mass for cathode and anode was taken according to the design of stoichiometric on the purpose of experiment. This electrode was dried at 120 °C under vacuum for 6 hrs to evaporate the water. The half-cells (CR 2032) were assembled in a glove box filled with dry Ar atmosphere. The electrolyte in the PC:EMC (3:7) was used. The concentration of the sodium salt was1 M $NaPF_6$. Electrochemical characterizations were conducted using in the galvanostatic charge-discharge tests (LAND 2001 CT battery tester). Cyclic voltammetry (CV) voltammograms were collected in the electrochemical workstation with the scanning rate of 0.1, 0.2, 0.4, 0.8 mV s^{-1}. SEM observations were carried out using an XL-30 microscope (Quanta FEG, FEI, PHILIPS). The residual pressure in the XPS analysis chamber was 9.3×10^{-7} Pa. To avoid any contamination from oxygen and water, the cycled electrodes were transferred from the Ar-filled glove box to the XPS chamber using a special air-proof device. In addition, the raw spectra were curve-fitted by non-linear least squares fittings with a Gauss–Lorentz ratio (80:20) using XPSPEAK41 software. XRD studies were carried out using a D8 Advance diffraction system (Bruker, Germany).

Figure. S1

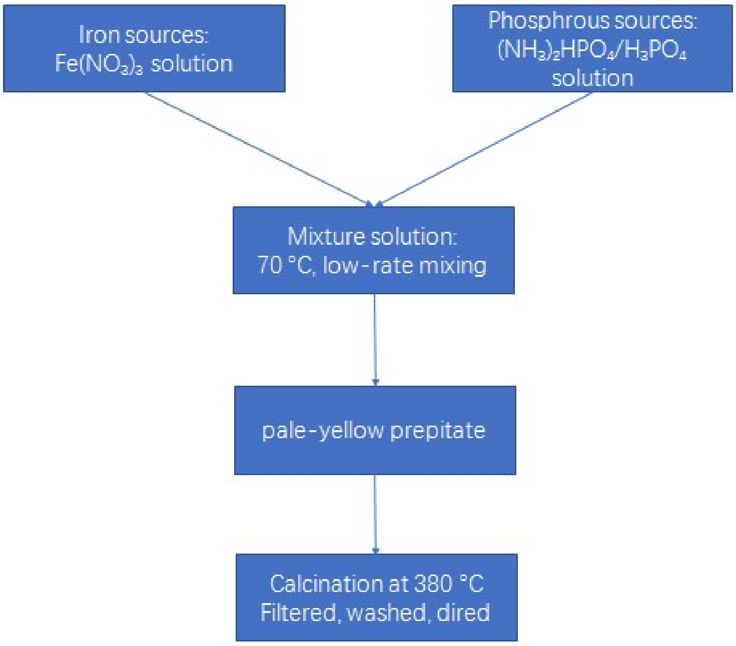

Figure. S2 XRD patterns were exhibited for both corresponding electrode.

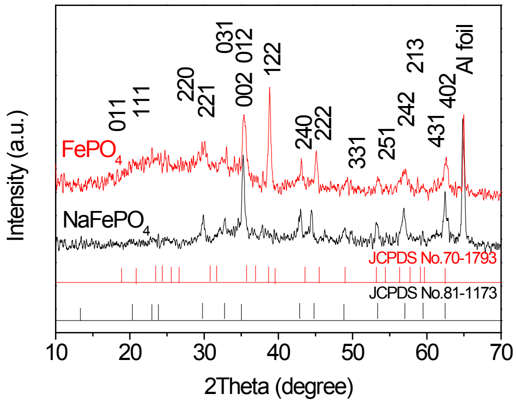
011
111
220
221
031
002 012
122
240
222
331
251
242
213
431
402
Al foil
FePO$_4$
NaFePO$_4$
JCPDS No.70-1793
JCPDS No.81-1173
Intensity (a.u.)
10
20
30
40
50
60
70
2Theta (degree)

Figure S3: *ex situ* XRD of graphite cathodes from (−)$FePO_4$/graphite(+) full cells being charged to different voltages, including OCV, 3 V, 3.5 V and 4 V.