Supplementary Information (SI) for ChemComm.

Supporting Information

Selective Lithium Extraction and Fe-P Component Reconstruction Toward High-Performance $Na_4Fe_3(PO_4)_2P_2O_7$ As Cathode Materials of Sodium Ion Batteries

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Experimental and characterization

Material preparation

The spent LFP cathode plates were collected from Shenzhen Hengchuang RuiNeng Technology Co., Ltd, China. The spent LFP cathode plates underwent thermal treatment in a tube furnace under argon atmosphere through a programmed heating regimen (5°C/min ramp rate to 400°C, 1 h dwell time), followed by ambient cooling and manual detachment of the recycled cathode active material (designated SLFP) from aluminum current collectors. Sodium persulfate ($Na_2S_2O_8$), sodium carbonate (Na_2CO_3), ammonium dihydrogen phosphate ($NH_4H_2PO_4$), sodium hydroxide(NaOH) and glucose ($C_6H_{12}O_6$) were supplied by Shanghai Macklin Biochemical Technology Co., Ltd. All chemicals used in this study were analytical grade reagents. Gases(SO_2) generated during the selective lithium extraction process are absorbed and treated using sodium hydroxide solution.

Characterization

X-ray diffraction (XRD) analysis was employed to investigate the crystallographic properties of the synthesized materials. The diffraction process involves coherent interference of X-ray waves scattered by the crystalline lattice, generating a distinct diffraction pattern through constructive and destructive wave interactions. Crystal structure parameters were derived through rigorous application of Bragg's law ($n\lambda = 2d \sin\theta$), enabling phase identification and structural characterization. Structural analysis was conducted using a Malvern Panalytical Empyrean 2 X-ray diffractometer equipped with Cu Kα radiation ($\lambda = 1.54184$ Å), operating at 40 kV and 40 mA. Scanning Electron Microscopy (SEM) was used to observe the microstructural morphology of the materials. A focused electron beam bombards the sample surface, where electron-sample interactions generate secondary electrons that are collected and converted into final images. Energy Dispersive Spectroscopy (EDS) attachment was simultaneously used to analyze the elemental distribution of the materials. The field-emission scanning electron microscope used in this study was sourced from TESCAN. X-ray photoelectron spectroscopy (XPS) was used to characterize the surface elemental composition, chemical states, and chemical environment of elements in materials. The XPS analyzer used in this study was the Thermo Scientific K-Alpha from the United States. Fourier-transform infrared spectroscopy (FTIR) was employed to characterize functional groups and molecular vibrations through mid-infrared absorption measurements (4000-400 cm^{-1}) with a spectral resolution of 4 cm^{-1}. The Fourier-transform infrared spectrometer used in this study was the Thermo Scientific IS50 model. Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) is a high-sensitivity and high-precision analytical technique for elemental analysis. This study employed an ICP-OES instrument (Model TCP-5100-VDV) manufactured by Agilent Technologies.

Electrochemical characterization

The active material, PVDF, and conductive carbon black were mixed and ground in a mass ratio of 8:1:1. N-methyl-2-pyrrolidone (NMP) was added dropwise and ground to form a homogeneous slurry. The slurry was then coated onto aluminum foil. The coated electrodes were vacuum-dried at 120°C for 8 hours to remove residual solvent. After drying, the electrodes were cut into 12 mm diameter circular cathodes. The cathodes and separators were vacuum-dried at 60°C for 4 hours before being transferred to an argon-filled glove box. The CR2032 button cell was assembled in an argon-filled glove box using an electrolyte of 1MLiPF6 in EC:DMC:DEC = 1:1:1 or 1M NaClO4 in EC:DMC = 1:1.

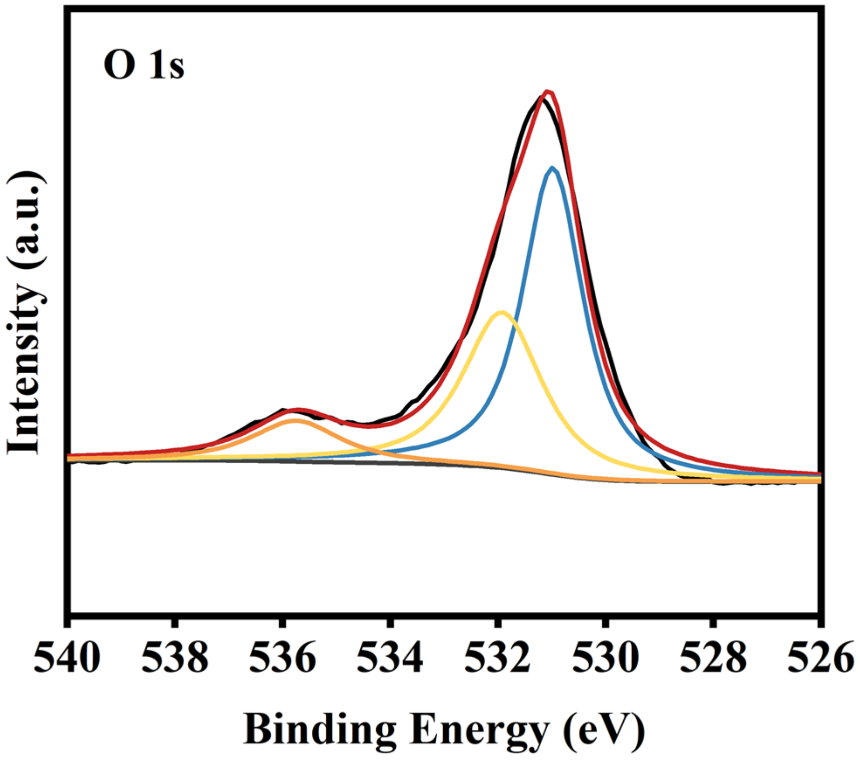

Figure S1. The O 1s spectrum of rNFPP.

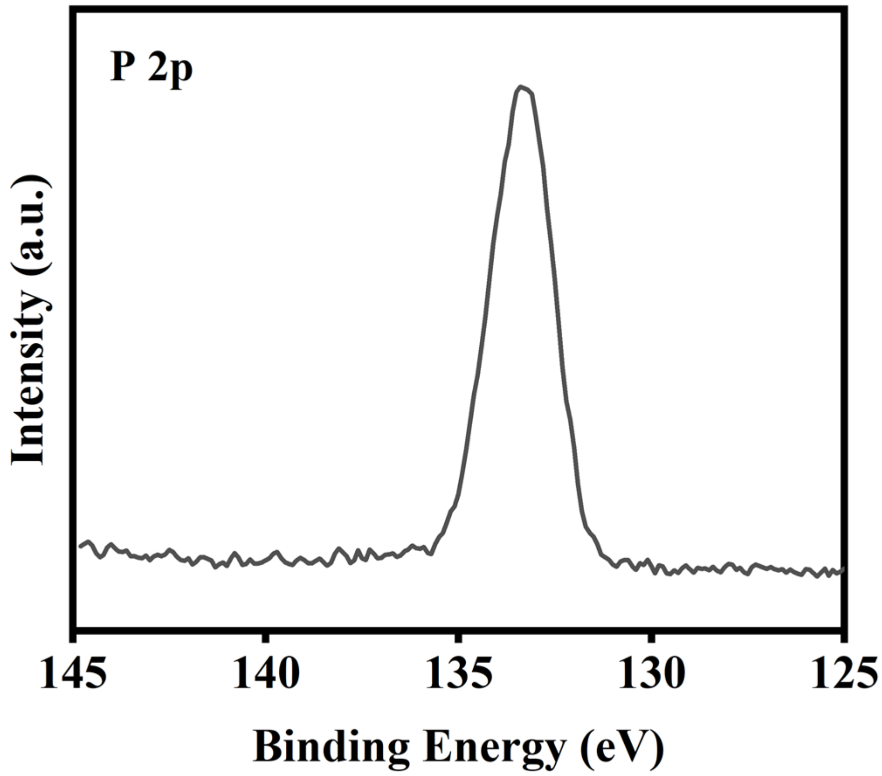

Figure S2. The P 2p spectrum of rNFPP.

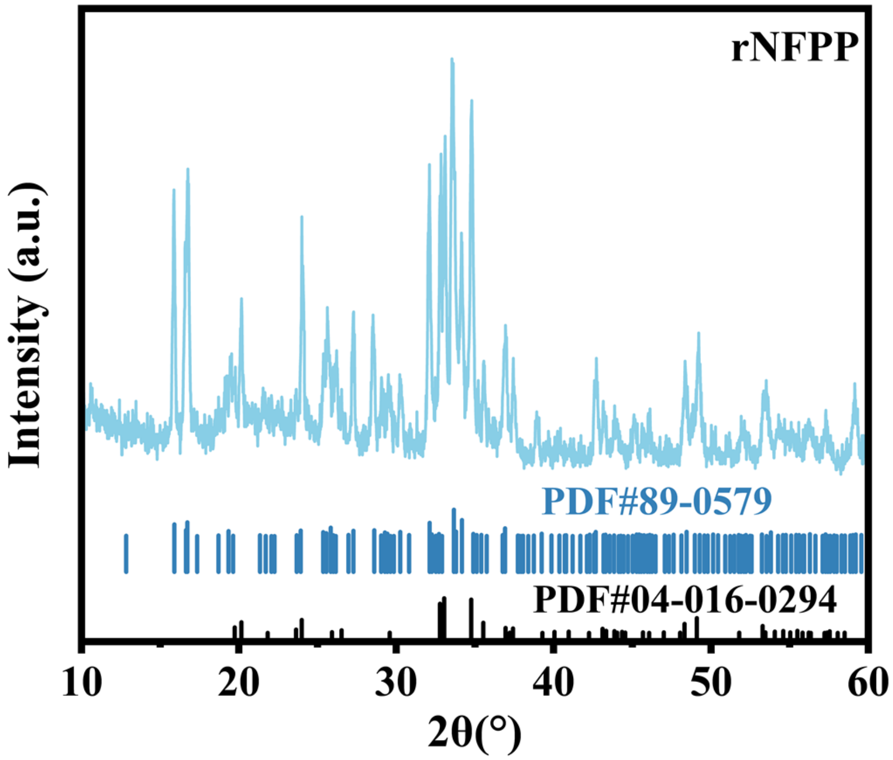

Figure S3. The XRD of rNFPP.

Figure S4. 5C cycle performance of SLFP.

Figure S5. 10C cycle performance of rNFPP.

Figure S6. 1C cycle performance of rNFPP at 50°C.

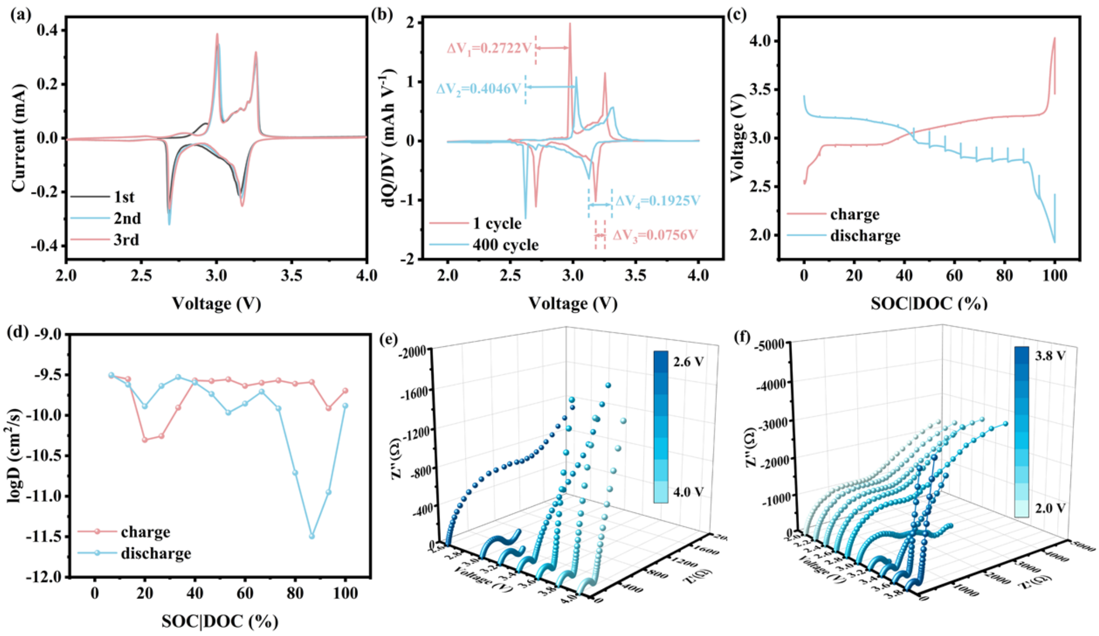

Figure S7. (a) The CV curves for rNFPP; (b) the dQ/dV curves for rNFPP; (c,d) The GITT curves and Na^+ diffusion coefficient curve for rNFPP; The in-situ electrochemical impedance spectroscopy for rNFPP (e) charge, (f) discharge.

Table S1 Main components of SLFP.

Content	Li	Fe	P	Al

Composition (wt%)	4.53	34.3	19.3	0.06

Table S2 Main components of SFP.

Content	Li	Fe	P
Composition (wt%)	0.08	35.51	20.32