



# Utilizing egg lecithin coating to improve the electrochemical performance of regenerated lithium iron phosphate



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## ABSTRACT

A series of egg lecithin coating LiFePO<sub>4</sub> composites have been prepared by a simple mechanochemistry activation and sintering processes using the regenerated LiFePO<sub>4</sub> as raw materials. The mechanical forces of high energy ball milling have induced activation or chemical changes of different components. The as-prepared LiFePO<sub>4</sub> composites showed stable (C+N+P) co-coating layers with N-C and P-C bonds wrapped LiFePO<sub>4</sub> particles and smaller uniform particle sizes, which can shorten the diffusion distance of lithium ions and electrons, and offer a good electronic conduction between the LiFePO<sub>4</sub> particles. The LiFePO<sub>4</sub>/C composite coating with 15% egg lecithin exhibited the average specific capacities of 164.9 mAh g<sup>-1</sup> at the low C-rate of 0.2 C; at C-rate of 5 C, the electrode can deliver the specific capacity of 120 mAh g<sup>-1</sup> with a capacity retention of 93% after 100 cycles; even at high rate of 20 C, this electrode materials can still keep 100.7 mAh g<sup>-1</sup>, which is 41% greater than its pristine counterpart. More interestingly, the raw material in this work is the recycled LiFePO<sub>4</sub> produced from the spent power batteries, which also demonstrates a greatly potential approach to utilize the regenerated LiFePO<sub>4</sub> from the spent batteries into high-performance cathode materials for Li-ion batteries applications.

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## 1. Introduction

Lithium iron phosphate (LiFePO<sub>4</sub>) is one of the most important cathode materials of rechargeable lithium ion batteries applied in many energy storage devices, due to its advantages of cost reduction, environmental benignity, electrochemical and thermal stability, appropriate operating voltage (3.45 V vs. Li/Li<sup>+</sup>), long cycle life (>2000 cycles) and relatively high theoretical specific capacity of 170 mAh g<sup>-1</sup> [1–6]. However, low electronic conductivity and lithium ion diffusivity are its biggest intrinsic drawbacks for commercial use as an advanced cathode material, which will restrict it from the large-scale applications in high-rate performance [7–10]. Especially, more and more recovered LiFePO<sub>4</sub> material will be reused with the development of the power batteries and electric vehicles. High-rate charge/discharge properties are more grievous limited due to the low electronic conductivity and the poor Li-ion diffusion efficiency.

In recent decades, many efforts have been performed to deal

with these issues of LiFePO<sub>4</sub>. The most effective strategy is to fabricate LiFePO<sub>4</sub> composites including optimizing particle size and their morphology [11,12], elements doping [13,14] and surface carbon coating [15–17]. Based on it, some LiFePO<sub>4</sub>/C hybrids have been developed, such as LiFePO<sub>4</sub>/mesoporous carbon [18], LiFePO<sub>4</sub>/graphene [19], LiFePO<sub>4</sub>/carbon nanowires [20], and LiFePO<sub>4</sub>/carbon nanotube [21], etc. Although the incorporation of carbonaceous matrix has significantly enhanced electrochemical performances, these LiFePO<sub>4</sub> composites are still confronted with limited rate-performance and reversible cycling life.

Recently, some researchers find that heteroatom (such as N, P, B and S) doped graphene crystal lattices can tailor both the chemical and physical reactivity as well as the electronic properties and it has been applied to significantly improve the electrode performance in LIBs [22,23]. The presence of heteroatoms at the carbon surface can effectively enhance the electric conductivity and the Li<sup>+</sup> storage capacity [22,24]. It suggests that nitrogen atom can act as an electron donor and provide electron carriers and reduce the band gap [25–29], resulting in the efficiently improved electrochemical performances. It is well known that P has higher electron-donating ability [30] although P and N are belong to the same group, so the phosphorus-doping can be expected to effectively improve the

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carbon conductivity since increasing electron-donating ability and the quantity of charge carriers comparing with that of carbon-containing materials [31–34].

Herein, we have prepared the (C+N+P) co-coating LiFePO<sub>4</sub> composites via simple mechanochemical ball-milling and calcination procedure using the recovered LiFePO<sub>4</sub> and egg lecithin as raw materials. It shows that N and P atoms have been introduced in the carbon layers via N-C and P-C bonds in the (C+N+P) co-coating LiFePO<sub>4</sub> composites. The as-prepared LiFePO<sub>4</sub> composites showed stable (C+N+P) co-coating layers wrapped LiFePO<sub>4</sub> particles and smaller uniform particle sizes, which can shorten the diffusion distance of lithium ions and electrons, and offer a good electronic conduction between the LiFePO<sub>4</sub> particles. The improved LiFePO<sub>4</sub>/C composite cathode exhibited the average specific capacities of 164 mAh g<sup>-1</sup> at the low C-rate of 0.2 C and the high capacity retention of 93% after 100 cycles at the rate of 5 C; even at high rate of 20 C, this electrode materials can still keep 100.7 mAh g<sup>-1</sup>. More interestingly, the recycled LiFePO<sub>4</sub>/C composite is produced from the spent LiFePO<sub>4</sub> power batteries. This work also demonstrates a greatly potential approach to utilize the regenerated LiFePO<sub>4</sub> from the spent batteries into high performance cathode materials for Li-ion batteries applications.

## 2. Experimental

### 2.1. Materials

Regenerated LiFePO<sub>4</sub> cathode materials from spent batteries were provided by GF Lithium Co. (Xinyu, China). Egg lecithin (EL) and N-methyl-pyrrolidinone was purchased from J&K Chemical. These reagents were used without further purification.

### 2.2. Preparation of samples

The (C+N+P) co-coating LiFePO<sub>4</sub> composite materials, LiFePO<sub>4</sub>/(C+N+P), were prepared via a simple mechanochemistry ball-milling and calcining process: Firstly, the regenerated LiFePO<sub>4</sub>/C as raw material was ground with egg lecithin, which was used as co-coating carbon, nitrogen and phosphorus sources. Then, the mixed slurry was dried in drying oven, and the final product was obtained after calcined at 600 °C for 4 h (with a heating rate of 5 °C min<sup>-1</sup>) under an Ar-H<sub>2</sub> (10%) flow in a tubular furnace. Next, the furnace was cooled down to room temperature under an Ar-H<sub>2</sub> (10%) flow. The (C+N+P) co-coating LiFePO<sub>4</sub> composite samples were obtained, which was denoted as LiFePO<sub>4</sub>/(C+N+P). To optimize the coating proportions, a serial of LiFePO<sub>4</sub>/(C+N+P) samples had been prepared according to different coating weight ratio of egg lecithin: 0 wt%, 5 wt%, 10 wt%, 15 wt%, 20 wt% and 25 wt%, respectively.

### 2.3. Structure and morphology characterizations

The as-prepared samples were characterized using X-ray diffraction (XRD, Panalytical) utilizing Cu K $\alpha$  radiation ( $\lambda = 0.154$  nm) at 40 kV and 30 mA, collecting data from 5° to 90° (2 $\theta$  degree) in the step of 2° min<sup>-1</sup>. SEM images were performed using FEI quanta 200 and HRTEM images were characterized by a high-resolution transmission electron microscope FEI Tecnai G2 F20. XPS measurements were performed by a PHI5000 Versa Probe spectrometer with a monochromatized Al K $\alpha$  X-ray source and an analyzer pass energy of 187.85 eV for survey scans.

### 2.4. Electrochemical characterization

Electrochemical measurements were carried out at room temperature using CR2025 coin-type cells with the lithium as the anode. The cathode was prepared using the mixture of active material as-prepared, acetylene black and polyvinylidene fluoride (PVDF) with a weight ratio of 80:10:10 in N-methyl-pyrrolidinone. Then the slurry was spread onto Al foil, followed by drying at 100 °C for 12 h in vacuum. The mass loading of LiFePO<sub>4</sub> cathode used in each cell was 2–2.5 mg cm<sup>-2</sup>. A mixed solvent of dimethyl carbonate (DMC)-ethylene carbonate (EC) containing 1.0 M LiPF<sub>6</sub> was mixed as the electrolyte. Coin-type cell was assembled in an argon-filled glove box, where the content of H<sub>2</sub>O and O<sub>2</sub> less than 0.5 ppm. The charging-discharging experiments were performed on the NEWARE battery testing system with the voltages ranging from 2.0 V to 4.2 V versus Li/Li<sup>+</sup> at room temperature. The electrochemical impedance spectroscopic analysis (EIS) was carried out with PARSTAT 2273 by using a 5 mV amplitude signal with the frequency range between 100 kHz and 100 mHz. Cyclic voltammograms analysis was performed by PARSTAT 2273 in the voltage area of 2.0–4.2 V at 0.1 mV s<sup>-1</sup>.

## 3. Results and discussion

### 3.1. Fabrication of (C+N+P) co-coating LiFePO<sub>4</sub> composites

Fig. 1 illustrated the fabrication process of (C+N+P) co-coating LiFePO<sub>4</sub> composite by a mechanochemistry activation and a simple sintering process. Typically, the regenerated LiFePO<sub>4</sub>/C as raw material was grind with egg lecithin by high energy ball-grinding, and the similar microcapsule composite of LiFePO<sub>4</sub>/(C+N+P) was formed. The mechanochemistry activation can be taken place in the ball-grinding process. It will induce some surface reactions and phase transformations between different components, and more stable composite formed. The egg lecithin was used as carbon, nitrogen and phosphorus source of (C+N+P) co-coating. Then, the final samples of LiFePO<sub>4</sub>/(C+N+P) was obtained after calcined at 600 °C in a tubular furnace. It is found that the as-prepared LiFePO<sub>4</sub> composites show smaller uniform particle sizes and uniform and

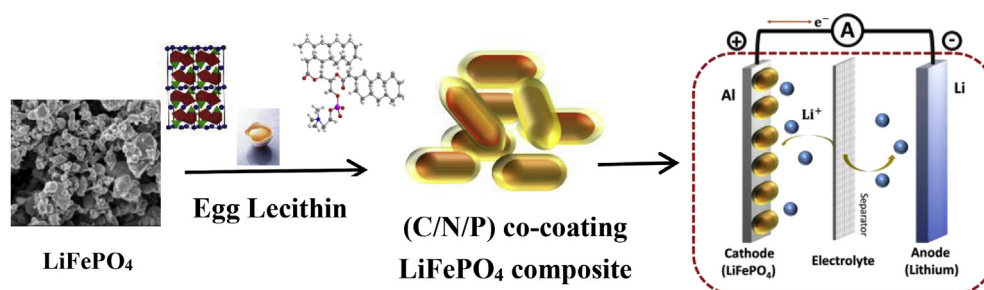


Fig. 1. Schematic illustration of (C+N+P) co-coating LiFePO<sub>4</sub> composite fabrication.

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