



Recovery of waste Li foils from spent experimental Li-anode coin cells for LiFePO₄/C cathode

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ABSTRACT

Waste Li foils in the spent experimental Li-coin-cells may bring the potential risk and the waste of Li-resource if they aren't reasonably treated in time. For this purpose, waste Li foils were recycled in the form of black LiFePO₄/C powders with the recovery of about 80% in this work. The obtained LiFePO₄/C powders were investigated in term of X-ray diffraction (XRD), scanning electron microscope (SEM), energy dispersive X-ray spectrum (EDX), inductively coupled plasma optical emission spectrometer (ICP-OES), transmission electron microscopy (TEM), nitrogen adsorption/desorption, galvanostatic charge/discharge, cyclic voltammetry (CV) and AC impedance spectroscopy. The results showed that black LiFePO₄/C powders consisted of many nano/micro particles with the specific surface area of 89.0 m²/g; furthermore, LiFePO₄/C cathode also delivered the satisfactory electrochemical performances. For example, the initial discharge capacity was 127.5 mAh/g and the reversible discharge capacity after 110 cycles was about 114.5 mAh/g at 0.1C. Seemingly, the satisfactory results may open a new avenue to develop the circular economy and the sustainable energy.

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1. Introduction

Currently, much attention has been paid to Li-air batteries and Li-S batteries owing to high theoretical specific capacity and energy density [1, 2]. In fact, these two kinds of batteries belong to Li anode batteries, in which metal Li foil acts as the anode. Although Li-air batteries and Li-S batteries are still in the early stage of the development in the labs, and the commercialization hasn't come true yet due to their low practical specific energy, the corresponding exploration and research in the labs have been in full swing, thus causing the accumulation of large quantity of spent experimental Li-anode batteries [3, 4]. Additionally, Li foils are also usually used as the counter/reference electrode in the experimental Li-coin-cells during the development of the new electrode active materials of lithium ion battery (LIB), and thus many spent many experimental Li-coin-cells were also accumulated in the labs. Obviously, these spent Li-anode batteries contain a lot of waste Li foils, resulting in the waste of Li resources and the potential risks to the environment if they are improperly treated. After multiple charge/discharge cycles, the resultant Li dendrites in the spent Li-anode batteries may pierce the separator film and result in the possible internal short-circuit or other calamities [5]. There may be also some potential explosions or fire hazard risks when these spent Li-anode batteries

are squeezed or heated, because Li metal with low melting point of only 180 °C is easily molten due to the inadvertent overheating [6]. Especially, Li-resource in the earth's crust is scarce and uneven. However, there are few reports on the recycling of waste Li foils from spent Li-anode batteries. Therefore, in order to alleviate the negative environmental effect and the imbalance between the large demand and the insufficient supply of Li-resource, it is very necessary to carry out the investigations about the recycling of waste Li foils from spent Li-anode batteries.

As we know, olivine LiFePO₄ has been widely considered as one of the most promising cathode active materials in LIB due to long flat charge/discharge plateau, high theoretical capacity (170 mAh/g), low cost, structure stability and environmental friendliness [7, 8]. However, a large quantity of Li resource need be consumed during the preparation of LiFePO₄. Herein, in order to alleviate the contradiction between high consumption and the shortage of Li resources, the new connection between waste Li foils and LiFePO₄ cathode need be built. In detail, waste Li foils were recycled in the form of black LiFePO₄/C powders from the spent experimental Li-coin-cells. The obtained LiFePO₄/C powders were investigated by methods of XRD, SEM, EDX, ICP-OES, TEM, nitrogen adsorption/desorption, galvanostatic charge/discharge, CV and AC impedance. Fortunately, LiFePO₄/C cathode exhibited the satisfactory electrochemical performances. Seemingly, a new avenue to develop the circular economy and the sustainable energy industry would be opened and launched.

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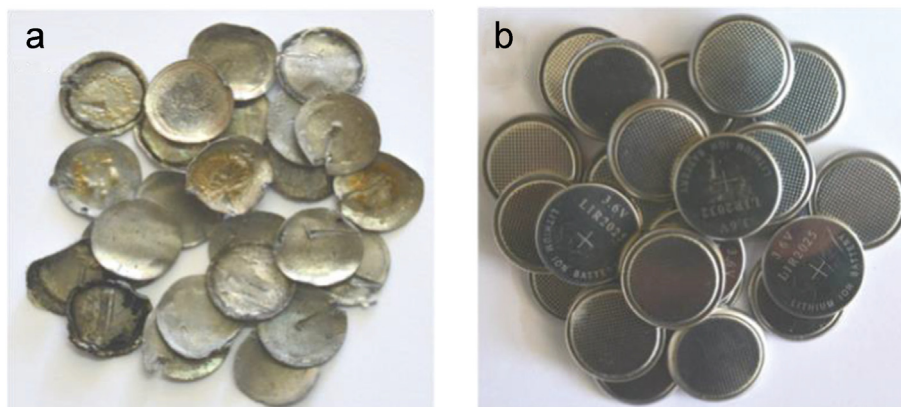


Fig. 1. (a) Waste Li foils and (b) spent experimental CR2025 Li-coin-cells.

2. Experimental

2.1. Materials

Waste Li foils (Fig. 1a) in this work were collected from spent experimental CR2025 Li-coin-cells in our lab (Fig. 1b), and these spent experimental Li-coin-cells were short-circuited or fully charged/discharged for at least 200 cycles.

2.2. Recovery of waste Li foils

Firstly, waste Li foils were obtained by manually dismantling some spent experimental Li-coin-cells and immediately weighted. Then, they were added into the deionized water for LiOH solution, which was mixed with H_3PO_4 , $\text{Fe}(\text{NO}_3)_3$ and citric acid ($\text{C}_6\text{H}_8\text{O}_7$), and the corresponding molar ratio was 2: 2: 2: 3. Then pH value of the mixture was adjusted to 7 with ammonium hydroxide, and 3% lauryl sodium sulfate (SDS) surfactant was added to the solution. The solution was heated and kept at $80\text{ }^\circ\text{C}$ under vigorously stirring until a yellowish green gel was formed, which was dried in a vacuum oven at $70\text{ }^\circ\text{C}$ for 12 h. Finally, the green dry gel was calcined at $700\text{ }^\circ\text{C}$ for 10 h in the tubular furnace with N_2 flowing.

2.3. Structure and morphology characterization

The morphology and microstructure were probed by XRD (TIRIII Rigaku Japan), SEM (Quanta 200 FEI USA) and TEM (JME-2100 JEOL Japan). The chemical component was characterized by ICP-OES (Prodigy Leeman USA) and EDX (Oxford instruments X-ray Microanalysis 1350). The corresponding surface area and pore volume were determined on the specific surface and pore size analysis instrument (Beishide 3H-2000PS1, China).

2.4. Electrochemical measurements

80 wt% LiFePO_4/C powders, 10 wt% acetylene black, and 10 wt% polyvinylidene fluoride binder (PVDF) were uniformly mixed in *N*-methyl-2-pyrrolidone for the electrode slurry. Then, LiFePO_4/C cathode was prepared by coating the electrode slurry on Fe foil current collector and dried at $60\text{ }^\circ\text{C}$ for 12 h, and the loading density of LiFePO_4/C was about $1.3\text{ mg}/\text{cm}^2$. The electrochemical performances of LiFePO_4/C cathode were measured by assembling CR2025 coin cells in an argon-filled glove box (Mikrouna Super 1220/750/900, China), in which Li foil was used as the counter/reference electrode, Celgard 2400 film was the separator, and the electrolyte was 1 M LiPF_6 in ethylene carbonate and



Fig. 2. Recycled procedures of waste Li foils from spent Li-foil-based coin cells.

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