Research Article

Excellent Temperature Performance of Spherical LiFePO_4/C Composites Modified with Composite Carbon and Metal Oxides

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Nanosized spherical LiFePO_4/C composite was synthesized from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Li}_2\text{C}_2\text{O}_4$, aluminum oxide, titanium oxide, oxalic acid, and sucrose by binary sintering process. The phases and morphologies of LiFePO_4/C were characterized using SEM, TEM, CV, EIS, EDS, and EDX as well as charging and discharging measurements. The results showed that the as-prepared LiFePO_4/C composite with good conductive webs from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ exhibits excellent electrochemical performances, delivering an initial discharge capacity of $161.7 \text{ mAh} \cdot \text{g}^{-1}$ at a 0.1 C rate, $152.4 \text{ mAh} \cdot \text{g}^{-1}$ at a 1 C rate and $131.7 \text{ mAh} \cdot \text{g}^{-1}$ at a 5 C rate, and the capacity retention of 99.1%, 98.7%, and 95.8%, respectively, after 50 cycles. Meanwhile, the high and low temperature performance is excellent for 18650 battery, maintaining capacity retention of 101.7%, 95.0%, 88.3%, and 79.3% at 55°C , 0°C , -10°C , and -20°C by comparison with that of room temperature (25°C) at the 0.5 C rate over a voltage range of 2.2 V to 3.6 V, respectively.

1. Introduction

Clean and rechargeable secondary energy sources are urgently needed for the development of modern technology and widespread application of portable electrical equipment. Among secondary energy sources, rechargeable lithium- (Li) ion battery is the most attractive and promising. In 1997, Padhi et al. [1] proved that olivine LiFePO_4 has an excellent performance in Li-ion intercalation and deintercalation. Its theoretical capacity is $170 \text{ mAh} \cdot \text{g}^{-1}$ with a flat discharge voltage at 3.4 V. Among polyanion cathodes, LiFePO_4 has the advantages of excellent cycle ability, good thermal stability, inexpensive raw materials, and environmental friendliness. LiFePO_4 is also attractive for use as a next generation cathode material for Li-ion batteries. However, the low electronic conductivity of LiFePO_4 leads to a poor charge-discharge performance at a high current rate. Therefore, modifications to LiFePO_4 to improve its properties have been developed. Such modifications include coating with nanocarbon [2–9], encapsulation with a conductive polymer [10, 11], and doping with suitable metals [12–18].

$\text{FePO}_4 \cdot x\text{H}_2\text{O}$ is a promising precursor for preparing the Li-ion battery cathode material LiFePO_4 . The advantages of

this precursor include innocuity, low cost, similar structure with LiFePO_4 , and oxidation avoidance of ferrous iron [19–21]. Traditional methods for the synthesis of $\text{FePO}_4 \cdot x\text{H}_2\text{O}$ and LiFePO_4 include solid phase synthesis [22, 23], sonochemistry [24], coprecipitations [25–29], and hydrothermal synthesis [30–32]. These methods usually involve the use of expensive metal-organic compounds. In the solid-state synthetic process, high-energy consumption is generated and particles are relatively unevenly distributed. The sonochemistry process has some advantages in preparing iron phosphate, including nonrequirement of oxidant, less reaction time, and controllable particle size. However, the large-scale production of iron phosphate is difficult to be realized [33, 34]. Lee and Teja [35] Xu et al. [36] prepared LiFePO_4 nanoparticles in subcritical and superheated water. Yu et al. [37] investigated the rapid and continuous production of LiFePO_4/C nanoparticles in superheated water. Chen et al. [38] reported the influences of carbon sources on the electrochemical performances of LiFePO_4/C composites.

In this work, nanosized spherical LiFePO_4/C composite was prepared from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Li}_2\text{C}_2\text{O}_4$, aluminum oxide, titanium oxide, oxalic acid, and

FIGURE 1: SEM images of nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ and LiFePO_4/C .

FIGURE 2: Schematic diagram of spherical LiFePO_4/C synthesized from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ (a) mixing process, (b) presintering stage and (c) sintered product.

FIGURE 3: TEM image of spherical LiFePO_4/C synthesized from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$.

sucrose by binary sintering process. The process is simple, requires uncomplicated equipment, and consumes low energy. Fine particles were obtained in homogenous distribution, appropriate for industrialized production.

2. Experimental

2.1. Preparation of Materials. The LiFePO_4/C composite was prepared by mixing stoichiometric amounts of nanosized

spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Li}_2\text{C}_2\text{O}_4$, aluminum oxide, and titanium oxide dispersed in ethylene glycol with oxalic acid and sucrose, followed by grinding via ball milling. After evaporating the ethylene glycol, the mixture was firstly sintered in a horizontal quartz tube at 400°C for 6 h in an argon atmosphere. As the presintered product cooled to room temperature, the LiFePO_4/C composites were obtained after being calcined at 650°C for 8 h.

2.2. Material Characterization. The LiFePO_4/C sample was characterized by X-ray diffraction (XRD; Rigaku D/MAX 2500 V) using $\text{CuK}\alpha$ radiation ($K\alpha = 0.154 \text{ nm}$) to identify the crystalline phase. Data were collected between 10° and 90° in steps of $8^\circ/\text{min}$. The surface morphologies of the samples were observed by a scanning electron microscopy (SEM) system (JSM-6380LV). The details of carbon coating were observed with transmission electron microscopy (TEM) (Hitachi, H-8100). A three-electrode system was used to characterize the electrochemical performances of the as-deposited LiFePO_4/C composites. LiFePO_4/C freshly deposited under the same experimental parameters were used in cyclic voltammetry (CV) analysis and normal charging and discharging experiments, as well as high rate charge and discharge characterizations. Electrochemical impedance spectroscopy (EIS) measurements were performed over a frequency range of 0.001 Hz to 1 MHz at a 50% discharge stage

FIGURE 4: EDS and EDX images of spherical LiFePO_4/C synthesized from spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$.

with a perturbation signal of 5 mV over a Chi 660c setup. All electrochemical measurements were conducted at room temperature (25°C).

2.3. Electrode Fabrication and Electrochemical Measurements.

The as-prepared cathode was mixed with acetylene black and polyvinylidene difluoride at a mass ratio of 80 : 10 : 10. LiFePO_4/C cathode was prepared by spreading the above mixture on an aluminum foil and drying in a vacuum oven at 120°C . Charge-discharge tests on LiFePO_4/C were performed in coin cells using LiFePO_4/C cathodes and Li anodes. A porous membrane (Celgard 2300) was used as a separator, and the electrolyte was $1\text{ mol}\cdot\text{L}^{-1}$ LiPF_6 dissolved in a mixture of ethylene carbonate, dimethyl carbonate, and methyl-ethyl carbonate at a volume ratio of 1 : 1 : 1. Coin cells (CR 2025) were assembled in an argon-filled glove box. The cells were charged and discharged at the rates of 0.1 C, 1 C and 5 C over a voltage range of 2.5 V to 4.2 V, respectively, versus the Li/Li^+ electrode at ambient temperature using a battery testing system (Neware BTS-2000). The high and low temperature performance of LiFePO_4/C was tested via fabricating 18650 battery at the rate of 0.5 C over a voltage range of 2.2 V to 3.6 V.

3. Results and Discussion

3.1. Morphology of LiFePO_4/C Prepared with Nanosized Spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$. Nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ has been used to prepare LiFePO_4/C . As seen in Figure 1, the SEM images of LiFePO_4/C from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ were shown. The products had diverse morphology. The as-prepared LiFePO_4/C composite was spherical and the particles' size close to 100 nm in size was uniformly distributed. The shapes of particles were traced back, following each step of preparation (Figure 2(a) for mixture, Figure 2(b) for presintering product, and Figure 2(c) for sintered product). Oxalic acid as reductant was ultimately decomposed into CO_2 and H_2O . Sucrose as carbon source was used for coating nanosized LiFePO_4 particles.

Figure 3 showed the TEM images of LiFePO_4/C . As shown in the image, LiFePO_4 particles were well surrounded by a thin surface layer of carbon. The thickness of the carbon-coated layer was about 4 nm. There was a layer of carbon web, providing good electronic contact between LiFePO_4 particles.

As seen from Figure 4, the EDS and EDX results showed that as-prepared sample had the elements, such as Fe, P, O, C, Ti, and Al, and all of the elements were distributed uniformly, indicating that C, Ti, and Al dispersed in LiFePO_4 evenly.

FIGURE 5: The initial charge/discharge curves and cycle performance.

FIGURE 6: The high and low temperature performance.

3.2. Electrochemical Performance of LiFePO_4/C Prepared with Nanosized Spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$

3.2.1. Initial Charge/Discharge Curves and Cycling Performance. The theoretical capacity of stoichiometric LiFePO_4 is $170 \text{ mAh} \cdot \text{g}^{-1}$. Figure 5 showed the initial charge-discharge capacity and cycling performance of the LiFePO_4/C composite cathodes at three different rates of 0.1 C, 1 C, and 5 C. The discharge capacity of LiFePO_4/C composite synthesized with nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ was 161.7, 152.4 and 131.7 mAh/g at 0.1 C, 1 C and 5 C, respectively. Noticeably, the long and flat voltage plateaus were at 3.44 and 3.40 V at the rate of 0.1 C for Li extraction and insertion, respectively. It implied the excellent two-phase redox reaction between FePO_4 and LiFePO_4 , as well as the typical electrochemical features of olivine-type LiFePO_4 . The small voltage difference between the charge and discharge plateaus at about 0.04 V was representative of good kinetics, especially considering

the poor electronic conductivity and low electrochemical diffusion rate of Li ions in a solid phase.

The cycle life of LiFePO_4/C composite synthesized from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ at the rates of 0.1 C, 1 C, and 5 C was shown. After 50 cycles, the capacity retention obtained 99.1%, 98.7%, and 95.8%, respectively.

3.2.2. High and Low Temperature Performance. The fatal disadvantage of commercial LiFePO_4 is that it is poor at low temperature performance. The general low temperature was referred to -20°C . Over a voltage range of 2.0 V to 3.65 V, commercial LiFePO_4 had a capacity retention ratio of 60%~70% at 0.5 C at -20°C by comparison with that of room temperature (25°C). Therefore, to resolve the problem plays an important role in industrialized application. The performance of LiFePO_4 can be characterized by 18650 battery with designed capacity of 1000 mAh. As illustrated in Figure 6, the high and low temperature performance of the LiFePO_4/C composite cathodes delivered the outstanding progress to the rate of 0.5 C over a voltage range of 2.2 V to 3.6 V for 18650 battery. Meanwhile, it was found that the battery exhibited a discharge of 1028.3 mAh, 1010.9 mAh, 960.4 mAh, 892.9 mAh, and 801.5 mAh at 55°C , 25°C , 0°C , -10°C , and -20°C , respectively. Compared with discharge performance at room temperature (25°C), the capacity retention maintained 101.7%, 95.0%, 88.3%, and 79.3% at 55°C , 0°C , -10°C , and -20°C , respectively. Moreover, the high discharge voltage plateaus at various temperatures demonstrated the as-prepared sample to be the excellent material for industrialized application.

3.2.3. CV and EIS. Freshly deposited LiFePO_4/C composite has been examined by CV at a scan rate of $0.1 \text{ mV} \cdot \text{s}^{-1}$, as shown in Figure 7. Figure 7(a) showed that the first CV curve of the LiFePO_4/C composite synthesized with nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ was described. The voltage difference was 0.19 V. The voltage observed at 3.52 V in the anodic sweep and at 3.33 V in the cathodic sweep characterized

FIGURE 7: The cyclic voltammogram curves and EIS plots.

the extraction and insertion of Li^+ in the LiFePO_4 olivine structure, respectively.

Meanwhile, as we can see from Figure 7(b), the electrochemical impedance of LiFePO_4/C synthesized from nano-sized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ was shown. The curve was formed by a depressed semicircle in the high- to middle-frequency region and a straight line in the low-frequency range. According to the literature [39], the depressed semicircle represented the charge-transfer reaction between the active materials and the electrolyte (R_{ct}). From the result, R_{ct} had the charge-transfer reaction resistance of 82Ω , indicating the excellent electrical conductivity and Li-ion diffusion of LiFePO_4/C composites.

4. Conclusions

LiFePO_4/C composites were then prepared from nanosized spherical $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Li}_2\text{C}_2\text{O}_4$, aluminum oxide, titanium oxide, oxalic acid, and sucrose by binary sintering process. The EDS and EDX showed that Ti and Al were distributed in LiFePO_4/C uniformly. And the composites had good conductive carbon webs and delivered an initial discharge capacity of $161.7 \text{ mAh} \cdot \text{g}^{-1}$ at a 0.1 C rate, $152.4 \text{ mAh} \cdot \text{g}^{-1}$ at a 1 C rate, and $131.7 \text{ mAh} \cdot \text{g}^{-1}$ at a 5 C rate. And the capacity retention obtained 99.1%, 98.7%, and 95.8%, respectively, after 50 cycles. Meanwhile, the high and low temperature performance is excellent for 18650 battery, exhibiting a discharge of 1028.3 mAh, 1010.9 mAh, 960.4 mAh, 892.9 mAh, and 801.5 mAh at 55°C , 25°C , 0°C , -10°C , and -20°C , respectively.

In summary, the as-prepared LiFePO_4/C materials had favorable properties for their commercial applications.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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