

Research Article

Influence of Temperature on the Performance of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ Prepared by High-Temperature Ball-Milling Method

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Aiming at the preparation of high electrochemical performance $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ cathode material for lithium-ion battery, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ was prepared with lithium carbonate, nickel (II) oxide, cobalt (II, III) oxide, and manganese dioxide as raw materials by high-temperature ball-milling method. Influence of ball-milling temperature was investigated in this work. It was shown that the fine $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder with high electrochemical performance can be produced by the high-temperature ball-milling process, and the optimal ball-milling temperature obtained in the current study was 750°C. Its initial discharge capacity was 146.0 mAhg⁻¹ at the rate of 0.1 C, and over 50 cycles its capacity retention rate was 90.2%.

1. Introduction

In the recent years, lithium-ion batteries have been widely used in small electronic devices because of their advantages in energy density and cycle life [1]. LiCoO_2 is generally employed as a cathode material, but co-bearing materials are expensive and could cause pollution issues. LiNiO_2 structure becomes unstable during the cell charging process as the material was delithiated. The instability of LiNiO_2 in lithiumion cells reduces the cycle life of the cell and leads to safety concerns [2]. Manganese cathode materials draw a lot of attention due to the advantages of low cost, abundant resources, and extensive distribution. LiMnO_2 is a kind of thermodynamic instable materials with high capacity. However, the layered structure is transformed into the spinel structure in the process of charge-discharge, leading to fast specific capacity attenuation and unstable electrochemical properties [3]. LiMn_2O_4 is prone to crystal transformation, solvation of manganese ion, and Jahn-Teller effect, leading to capacity fade in lithium batteries [4]. LiFeO_4 is regarded to be the zero pollution anode material, which has the advantage of cheap price and high security, but its application is limited due to the low conductivity and tap density [5, 6].

The layered ternary material which was first reported by Ohzuku and Makimura [7] cumulates the merits of LiCoO_2 , LiNiO_2 , and LiMnO_2 . It is considered as one of the promising anode materials on account of high discharge specific capacity, static cycle life, low cost, and good safety performance [8].

Current methods for the large-scale preparation of high-performance cathode materials of three elements mainly include coprecipitation method [9–11] and solid-phase method [12–14]. The material synthesized by the solid-phase method has low impurity and capacity, while coprecipitation method could solve this problem. Some approaches such as sol-gel method, [15–17], microemulsion method [18], microwave method [19], spray pyrolysis [20], and combustion method [21] were studied in lab scale to prepare high-performance materials, but all these approaches are usually of high costs, have complicated processes, and are not environment-friendly.

High-temperature ball-milling method was modified from a traditional planetary mill by equipping controllable heating and automatic temperature-controlled elements; so, milling and sintering treatments could be simultaneously conducted in one reactor shortening the synthetic time [22–24]. In this work, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was prepared by high-temperature

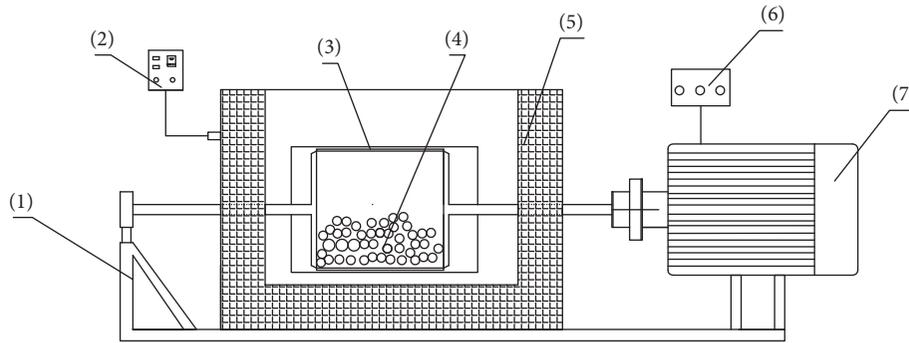


FIGURE 1: Illustration of the high-temperature ball mill. (1) Support base, (2) temperature controller, (3) ball-milling canister, (4) grinding ball, (5) furnace body, (6) speed regulator, and (7) electrical motor.

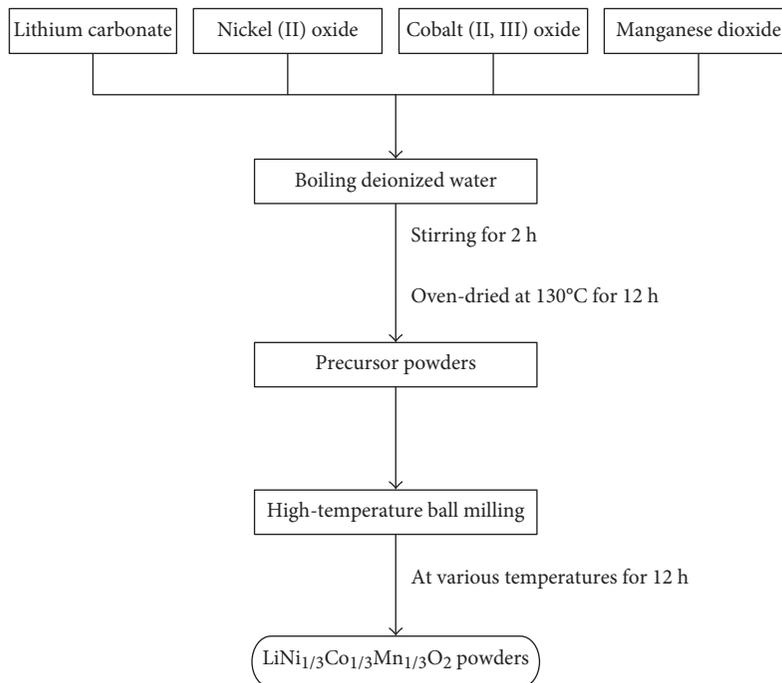


FIGURE 2: Flow sheet of the process route.

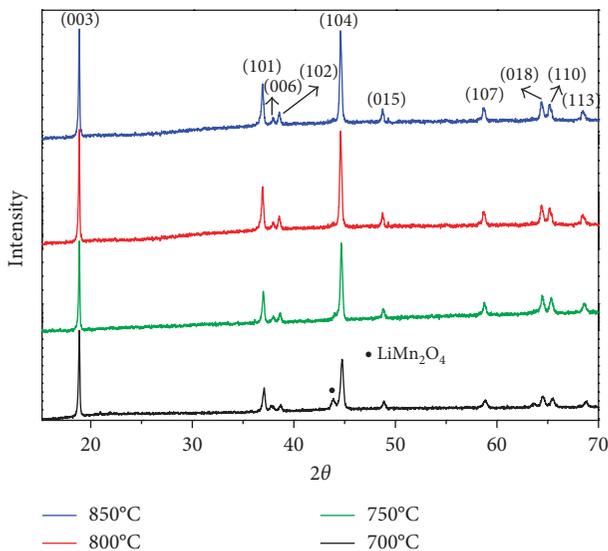


FIGURE 3: XRD patterns of products prepared at various temperatures.

TABLE 1: Lattice parameter of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powders prepared at various temperatures.

N	a -axis (\AA)	c -axis (\AA)	c/a ratio
700°C	2.8642	14.1895	4.954
750°C	2.8653	14.2384	4.969
800°C	2.8661	14.2363	4.967
850°C	2.8667	14.2295	4.964

ball-milling method with Li_2CO_3 , NiO , Co_3O_4 , and MnO_2 as raw materials, and the influence of milling temperature (700, 750, 800, and 850°C) on the product was investigated.

2. Experimental

A mass of 39.365 g lithium carbonate (Li_2CO_3 , AR, 99.9%), 25.885 g nickel (II) oxide (NiO , CP, 99.5%), 27.677 g cobalt

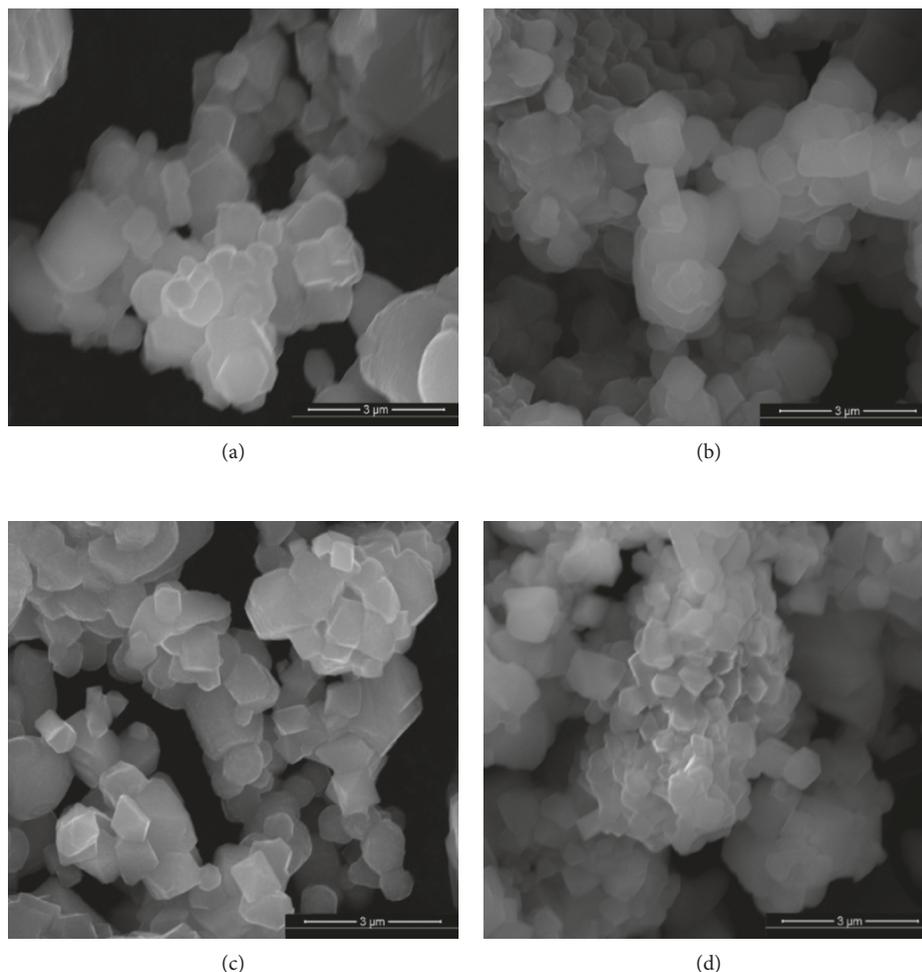


FIGURE 4: SEM images of products prepared at various temperatures of (a) 700°C, (b) 750°C, (c) 800°C, and (d) 850°C.

(II, III) oxide (Co_3O_4 , AR, 99.9%), and 29.977 g manganese dioxide (MnO_2 , AR, 99.9%) were dispersed in 250 mL boiling deionized water, which was agitated for 2 h at room temperature for intensive mixing. The precursor for $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ was obtained after drying the product slurry at 120°C for 12 h. The HTBM was conducted in the novel mill, as illustrated in Figure 1(a). The precursor was poured into a jar with some milling balls at a ball-to-powder mass ratio of 15 : 1 followed by sealing of the mill [24]. The samples were heated to the set temperatures (700, 750, 800, and 850°C) and held at these temperatures for 12 h. A flow sheet of the process route is given in Figure 2.

The structure of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ was determined by X-ray diffraction (XRD) (Rigaku D/max-RB) with Cu-K α radiation. The analysis was performed between 10° and 70° at a scan rate of 0.02°/min. The morphology and microstructure of the sample were characterized using scanning electron microscopy (SEM, HITACHI S-3000H) analysis.

The electrochemical performances of the samples were investigated using a two-electrode coin cell (CR2032). To prepare the working electrodes, 80% of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder, 10% of acetylene black, and 10% of polyvinylidene fluoride (PVDF) were dissolved in *N*-methyl pyrrolidone (NMP), and the electrode slurry was coated on an Al foil (the

average weight of the active material in per piece of electrode was 6.7 mg) and then heated at 120°C under vacuum for 12 h. 1 M LiPF_6 in a mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC) (1 : 1 in volume) was used as liquid electrolyte. Button batteries were assembled in argon-filled glove box with lithium metal as a counterelectrode and polypropylene (Celgard 2400) as a separator. Cycle charge-discharge tests at different rates were conducted using a NEWARE battery-testing system (Neware Co., Ltd., China) from 2.5 to 4.3 V at the room temperature (the current of 1 C equals to 200 mAhg⁻¹).

3. Results and Discussion

The XRD patterns of the resulting $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ samples synthesized at different temperatures of 700, 750, 800, and 850°C for 12 h are shown in Figure 3. The diffraction peak intensity of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ was enhanced with the rise of ball-milling temperature, demonstrating that temperature has a great influence on the crystallinity of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$. When the temperature was 700°C, some LiMn_2O_4 was generated, which is regarded as an impurity, showing that the reaction did not go completely. Diffraction peaks show a single phase of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ in an α -layered structure

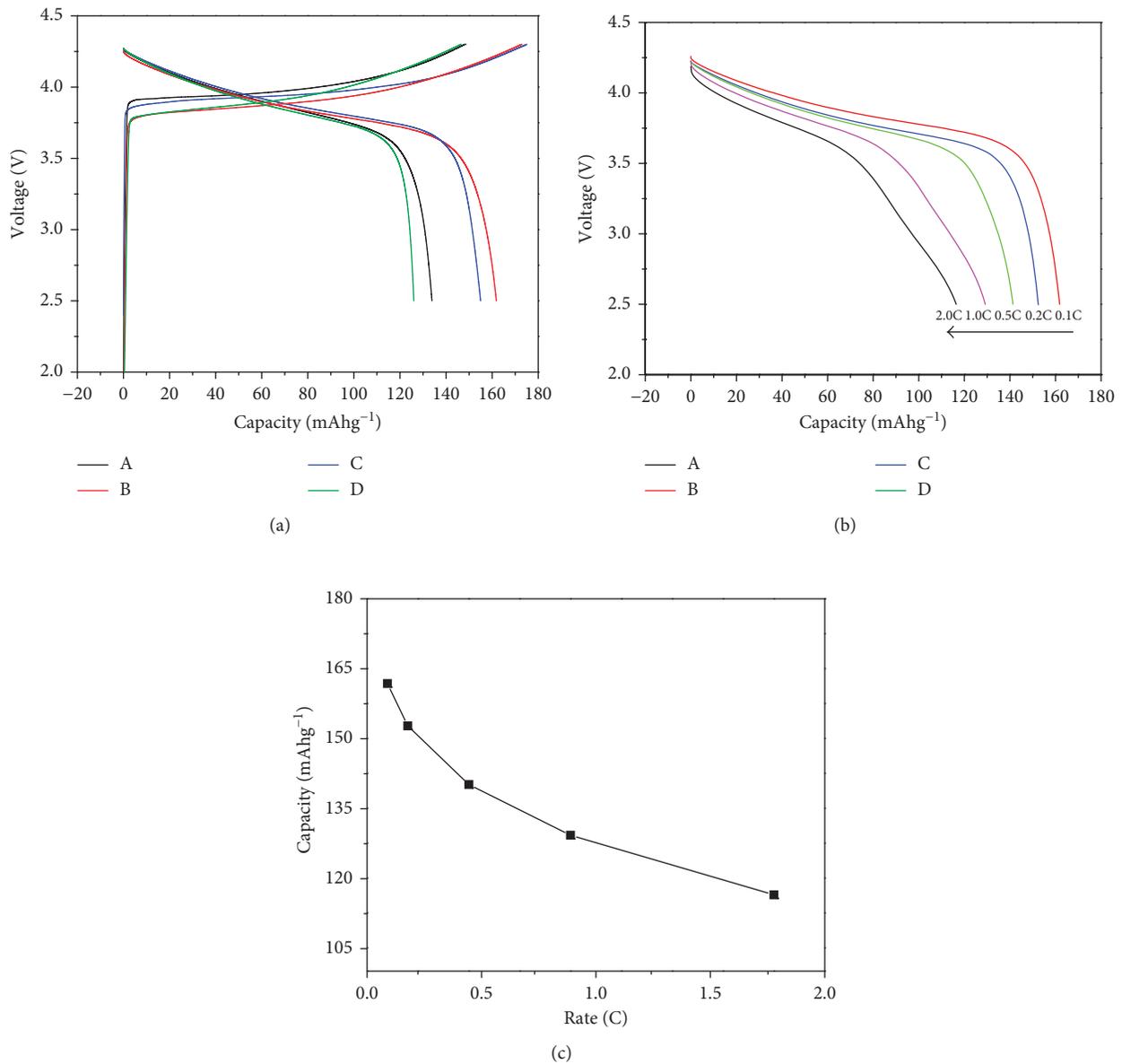


FIGURE 5: (a) Charge-discharge curves of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powders prepared at various temperatures: (A) 700°C, (B) 750°C, (C) 800°C, and (D) 850°C at 0.1 C rate, (b) discharge curves at various currents, and (c) the dependence of specific capacity on current density.

without any impurity phase when the temperature range is 750~850°C.

$\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 700°C had unit cell parameters (Table 1) of $a = 2.8642 \text{ \AA}$ and $c = 14.18953 \text{ \AA}$. $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 750°C had unit cell parameters of $a = 2.8653 \text{ \AA}$ and $c = 14.2384 \text{ \AA}$. $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 800°C had unit cell parameters (Table 1) of $a = 2.8661 \text{ \AA}$ and $c = 14.2363 \text{ \AA}$. $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 850°C had unit cell parameters (Table 1) of $a = 2.8667 \text{ \AA}$ and $c = 14.2295 \text{ \AA}$. The ratio of c/a is usually used as a measurement of hexagonality for layered materials. When the ratio is greater than 4.9, it is assumed that the material possesses layered characteristics [25]. Table 1 shows that c/a of all the samples are greater than 4.9; it means all the samples possess layered characteristics.

SEM images of the high-temperature ball-milling products prepared at different temperatures for 12 h are shown in Figure 4. It can be clearly found that some large particles were formed with the increase of temperature. When the temperature was 700°C, the product particles were small but in a severe agglomeration. When the temperature rose to 750°C, smaller spherical particles around 2–4 μm in length are obtained and the agglomeration was reduced. The grain sizes of the particles notably increased, and the agglomeration became more serious when the ball-milling process was conducted at 800 and 850°C.

The electrode characteristics of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at various ball-milling temperatures were examined by galvanostatic charge-discharge tests. The cell was cycled between 2.5 and 4.3 V using 0.1 C rate at room

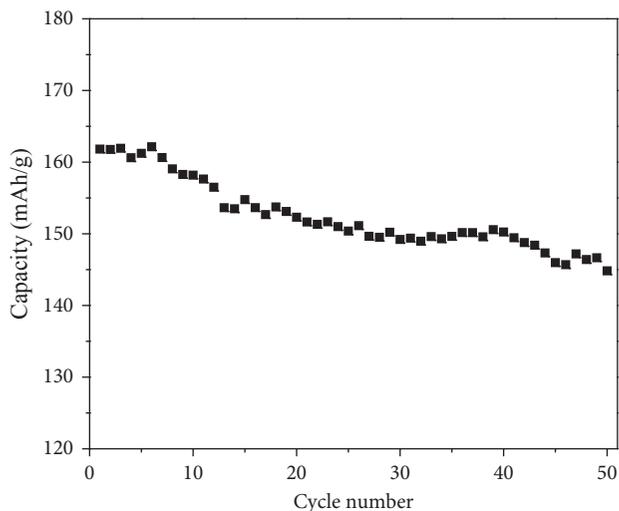


FIGURE 6: The cyclic performance at a current of 0.1 C of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 750°C .

temperature. Figure 5(a) shows the initial charge-discharge curves of the four samples. Experimental results indicated that initial discharge capacities are 133.7, 161.8, 155.0, and 126.1 mAhg^{-1} , respectively, and the sample synthesized at 750°C has the highest capacity. The reason of this phenomenon include the following several aspects: some impurity of LiMn_2O_4 was generated when ball-milling process was carried out at 700°C , and when ball-milling temperatures were rose to 800 and 850°C , the grain sizes of the powder increased and the crystal agglomeration became serious. Therefore, the optimal ball-milling temperature for preparing $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder obtained in the current study was 750°C .

The rate performance at different currents of the $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powders prepared at 750°C is shown in Figure 5(b). Accordingly, the dependence of specific capacity on current density is given in Figure 5(c). The initial discharge capacities of the sample were 161.8, 152.7, 140.1, 129.2, and 116.4 mAhg^{-1} , at rates of 0.1, 0.2, 0.5, 1.0, and 2.0 C, respectively. When currents increased, the discharge capacities of the sample were slightly reduced. The initial discharge capacity was 116.4 mAhg^{-1} at the rate of 2.0 C, demonstrating that the $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder synthesized at 750°C had an excellent rate performance. The cyclic performance at the current of 0.1 C of the $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared at 750°C is shown in Figure 6. After 50 cycles, the capacity of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was 146.0 mAhg^{-1} ; the capacity retention rate achieved 90.2% showing an excellent rate cyclic performance.

It is reported that $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder had been synthesized by many methods. $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was prepared at 900°C for 12 h by coprecipitation method [26]; $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was obtained at 500°C for 6 h and then at 900°C for 12 h by original wet-chemical route [27]; $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was synthesized at 800°C for 20 h via microwave-hydrothermal method [28]. In this work, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder with performance uniformity was synthesized at a ball-milling temperature of

750°C for 12 h. Its initial discharge specific capacity was 161.8 mAh/g at the rate of 0.1 C from 2.5 V to 4.3 V. It means that $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder synthesized at 750°C for 12 h by high-temperature ball-milling method had an excellent electrochemical performance.

4. Conclusions

In this work, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was prepared via high-temperature ball-milling method, and the effect of temperature on the $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder was investigated. Results showed that the optimal ball-milling temperature for preparing $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder obtained in the current study is 750°C . The products have an excellent electrochemical performance.

Compared to other methods, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder prepared by high-temperature ball-milling method needs lower temperature. Based on the findings of this study, it is suggested that preparation of $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ powder by high-temperature ball milling is simple for equipment, suitable for mass production, and easy for realization of industrialization.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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