

## Research Article

# Zn-Doped $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ Composite as Cathode Material for Lithium Ion Battery: Preparation, Characterization, and Electrochemical Properties

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Zn-doped  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  composite,  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ( $x = 0.02; 0.05; 0.08$ ), is synthesized by the sol-gel method. The crystal structure, morphology, and electrochemical performance are investigated via X-ray diffraction (XRD), scanning electron microscope (SEM), cyclic voltammetry (CV), and constant current charge/discharge experiment. The result reveals that Zn-doping cathode material can reach the initial charge/discharge capacity of  $188.8/162.9 \text{ mAh}\cdot\text{g}^{-1}$  for  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  and  $179.0/154.1 \text{ mAh}\cdot\text{g}^{-1}$  for  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.95}\text{Zn}_{0.05}\text{O}_2$  with the high voltage of 4.4 V at 0.1 C. Furthermore, the capacity retention of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  is 95.1% at 0.5 C after 50 cycles at room temperature. The improved electrochemical properties of Zn-doped  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  are attributed to reduced electrode polarization, enhanced capacity reversibility, and excellent cyclic performance.

## 1. Introduction

Nowadays, the lithium ion battery as a fairly new member in the battery technology has been widely studied due to its promising applications in the field of vehicle, military, space, and medical devices [1]. Of the reported lithium based cathode materials, the  $\text{LiCoO}_2$  has excellent discharge capacity and cyclic performance and is still the most popular candidate for the lithium ion battery application. However, the toxicity and high cost of  $\text{LiCoO}_2$  have restricted its further practical utilization. Recently, the  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  has attracted intensive attentions as an ideal alternative cathode material candidate to the traditional  $\text{LiCoO}_2$  [2]. The layered  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  has a typical hexagonal  $\alpha\text{-NaFeO}_2$  structure with a space group of R3m. Moreover, the  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  held some prominent advantages, such as high capacity, thermal stability, safety, and low cost [3], which make it as a promising cathode material for high power application, for instance, the electric vehicle and portable device. But there still exist two shortcomings limiting further application of  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  in lithium ion battery.

One is the poor lithium ion diffusion efficiency that would cause low electronic conductivity [4]; another is the severe capacity decay, especially working under the high voltage [5]. Doping the parent  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  with another transit metal has been regarded as an effective approach to improve the electrochemical properties of cathode materials. So far, some transition metals such as La [6] and Ti [7] or nontransition metals such as Al [8] and Mg [9] have been reported as the doping element for the  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ . It is found that the doping can avoid the unfavorable cation mixing of active materials under high voltage.

Different from the typical synthesized method, including coprecipitation method [10], solid-state method [11], and hydrothermal method [12], herein, the Zn-doped  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  is prepared by a facile sol-gel method, with purpose to modify the electrochemical properties of this cathode materials. The crystal structure, morphology and electrochemical performance are investigated via X-ray diffraction (XRD), scanning electron microscope (SEM), cyclic voltammetry (CV), and constant current charge/discharge experiment.

## 2. Experimental

**2.1. Preparation of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  Samples.** All chemicals (analytical grade or better) were purchased commercially and used without any further purification. The  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  cathode material was synthesized by sol-gel method. Metal precursors, including  $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{CH}_3\text{COOLi} \cdot 2\text{H}_2\text{O}$ , and  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ , were stoichiometrically mixed ( $n(\text{Li}^+) : n(\text{Ni}^{2+} + \text{Co}^{2+} + \text{Mn}^{2+}) : n(\text{Zn}^{2+}) = 1 : (1-x) : x$  ( $x = 0.02; 0.05; 0.08$ )) as raw materials;  $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$  was used as complexing agent. Firstly, the mixture was dissolved into aqueous solutions and then the  $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$  was dropped slowly into the aqueous solution under stirring at  $100^\circ\text{C}$  oil bath. The mixtures were kept under vigorous stirring to obtain the gel-like form. The wet gel was dried at  $100^\circ\text{C}$  for 24 h to get an amaranth dry gel and then ground into powder. Finally, the powder was preheated at  $400^\circ\text{C}$  for 3 h and then calcined at  $800^\circ\text{C}$  for 10 h in a muffle.

**2.2. Characterization and Electrochemical Measurement.** The crystallinity and structure of the samples was characterized by X-ray diffraction (XRD, D-MAY iiA, Japan) with  $\text{Cu K}\alpha$  radiation. The morphology of synthesized products was observed on a scanning electron microscopy (SEM, Hitachi, S-3400N, Japan).

The electrochemical characteristics of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  samples were investigated using CR2430 coin cells assembled in an argon-filled glove box. To prepare the working electrode, the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$ , acetylene black, and polyvinylidene fluoride (PVDF) with a weight ratio of 8 : 1 : 1 were mixed in N-methylpyrrolidone (NMP) to obtain a homogeneous slurry, which was spread on the Al foil. A metallic lithium foil was used as the counter and reference electrode. The celgard 2400 polypropylene porous film served as the separator and electrolyte consisted of a solution of 1 M  $\text{LiPF}_6$  in ethylene carbonate (EC) and dimethyl carbonate (DMC) (1 : 1 in volume).

The assembled cells were charged and discharged with constant current on the NEWARE battery program-control test system (Shenzhen, China) in a potential range of 2.8–4.4 V (versus  $\text{Li}^+/\text{Li}$ ) at room temperature ( $25^\circ\text{C}$ ).

The cyclic voltammetry was performed on CHI660C electrochemical workstation (Wuhan, China) between 2.5 and 4.5 V at room temperature, at the scanning rate of  $0.1 \text{ mVs}^{-1}$ .

## 3. Results and Discussion

**3.1. Characterization of Zn-Doped  $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$  Composite.** The  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  samples were synthesized with different doping content of Zn, so they are referred to as Zn-X. Figure 1 shows the XRD patterns of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$ . All the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  samples have a typical hexagonal  $\alpha\text{-NaFeO}_2$  structure (JCPDS card number 50-0653); no diffraction peaks of impurity can be

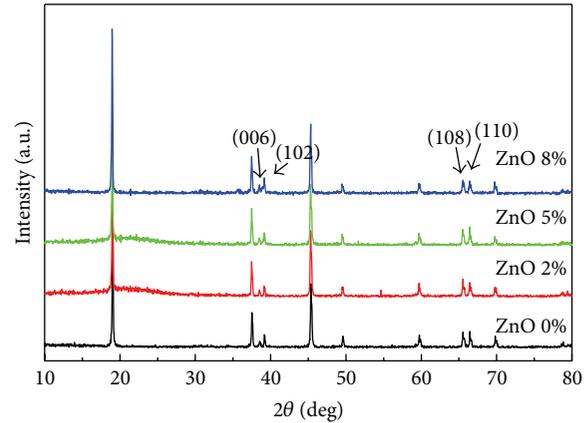


FIGURE 1: XRD patterns of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  sample doping with different Zn contents.

TABLE 1: Lattice parameters of samples doping with different content of Zn.

Sample	Lattice parameters			$I_{003}/I_{104}$
	$a/\text{\AA}$	$c/\text{\AA}$	$c/a$	
$\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$	2.8532	14.1653	4.9647	1.482
$x = 0.02$	2.8516	14.1755	4.9711	1.473
$x = 0.05$	2.8593	14.2062	4.9684	1.431
$x = 0.08$	2.8601	14.2273	4.9744	1.393

found, revealing that partial substitution of Zn in the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  cannot change the crystal structure. This result could indicate that the Zn element is totally inserted into the lattice of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$ . Moreover, the diffraction patterns show clear splitting of the hexagonal characteristic doublets of (006)/(102) and (108)/(110); this can be ascribed to the layered structure of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  [13]. Table 1 gives the refined lattice parameters of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$ . The lattice expansions are slightly increased with Zn-doped content, which further illustrates that the  $\text{Zn}^{2+}$  ions have doped into the lattice of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  during the calcined process. Since the metal ions, for example,  $\text{Ni}^{2+}$  (0.069 nm),  $\text{Co}^{3+}$  (0.0545 nm), and  $\text{Mn}^{4+}$  (0.054 nm), have smaller ion radius than that of  $\text{Zn}^{2+}$ , when they are replaced by the  $\text{Zn}^{2+}$  (0.074 nm),  $\text{Zn}^{2+}$  ions would enlarge the lattice parameter. Similar results have observed on the effect of Mg doping into lithium nickel cobalt oxides by Pouillier et al. [14]. Besides, the values of  $I(003)/I(104)$  are  $\geq 1.2$ , indicating that the samples have low cation mixing [15]. The cation mixing will lead to an increase of disorder, making an undesirable electrochemical performance of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$ , for example, low lithium conductivity, low capacity, and poor cyclic performance [4].

The morphologies of the layered composites before and after Zn modification are shown in Figure 2. It is found that the Zn-doping does not change their morphologies. The bare and modified materials are uniformly distributed with particles size of 200–300 nm, which would facilitate the

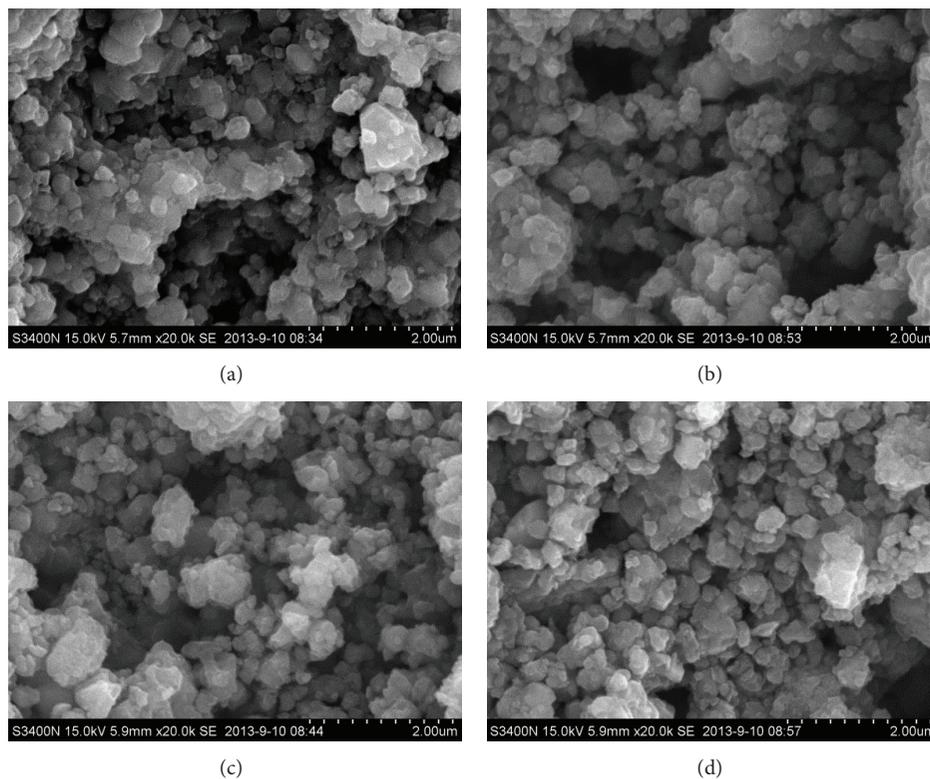


FIGURE 2: SEM images of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  powders ((a) 0; (b) 2%; (c) 5%; (d) 8%).

intercalate and deintercalate process for  $\text{Li}^+$  and improve ion conductivity and rate performance.

**3.2. Electrochemical Performances.** The galvanostatic charge/discharge tests were conducted to study the electrochemical performances. Figure 3 shows the initial charge/discharge curves of the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ( $x = 0, 0.02, 0.05, 0.08$ ) with the current rates of 0.1C between 2.8 V and 4.4 V at room temperature. Increasing the Zn-doping content, the charge/discharge capacities declined slightly, which can be ascribed to the fact that the  $\text{Zn}^{2+}$  with larger radius block the  $\text{Li}^+$  intercalation-deintercalation path or decrease volume concentration of  $\text{Ni}^{2+}$  [16]. The charge/discharge capacities are 195.4/168.7, 188.8/162.9, 179.0/154.1, and 177.7/142.3  $\text{mAh}\cdot\text{g}^{-1}$  for Zn-0, Zn-2, Zn-5, and Zn-8, respectively. Correspondingly, the irreversible capacities are 26.7, 25.9, 24.9, and 35.4  $\text{mAh}\cdot\text{g}^{-1}$ , respectively. Though Zn-doping decreases the charge/discharge capacities, Zn-2 and Zn-5 show greater efficiency than Zn-0, owing to the Zn-doping. Therefore, the capacity retention of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  cathode material at high voltage (4.4 V) is improved.

Cycling performance is one of the significant electrochemical characteristics of lithium ion battery for high-voltage application. Figure 4 shows the relationship of discharge capacities and cycle times at different discharge rates. As shown from Figure 4, owing to the increase of discharge current, the capacity of discharge declines slightly during the cycling process. The initial

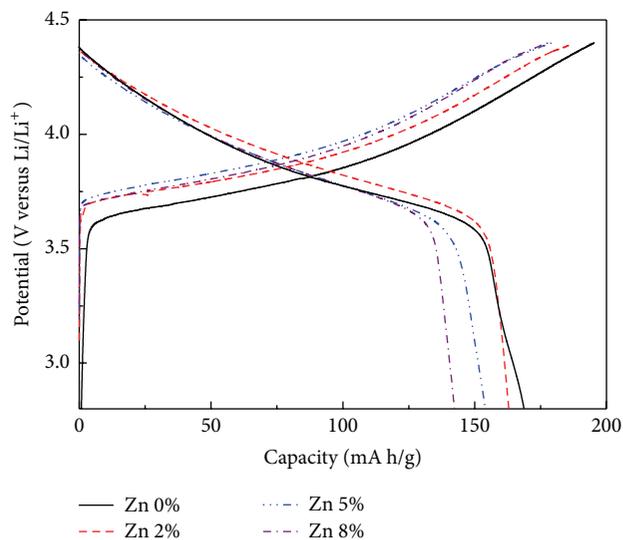


FIGURE 3: Initial charge/discharge curves of Zn-doping samples at 0.1C.

discharge capacity of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  is  $163.6 \text{mAh}\cdot\text{g}^{-1}$ ; additionally, the discharge capacities of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  maintain  $157.7 \text{mAh}\cdot\text{g}^{-1}$  at 0.5C after 20 cycles, and the capacity retention is 96.4%. After 40 cycles, the discharge capacity declines by  $7 \text{mAh}\cdot\text{g}^{-1}$ , and the capacity retention is 95.7%. At

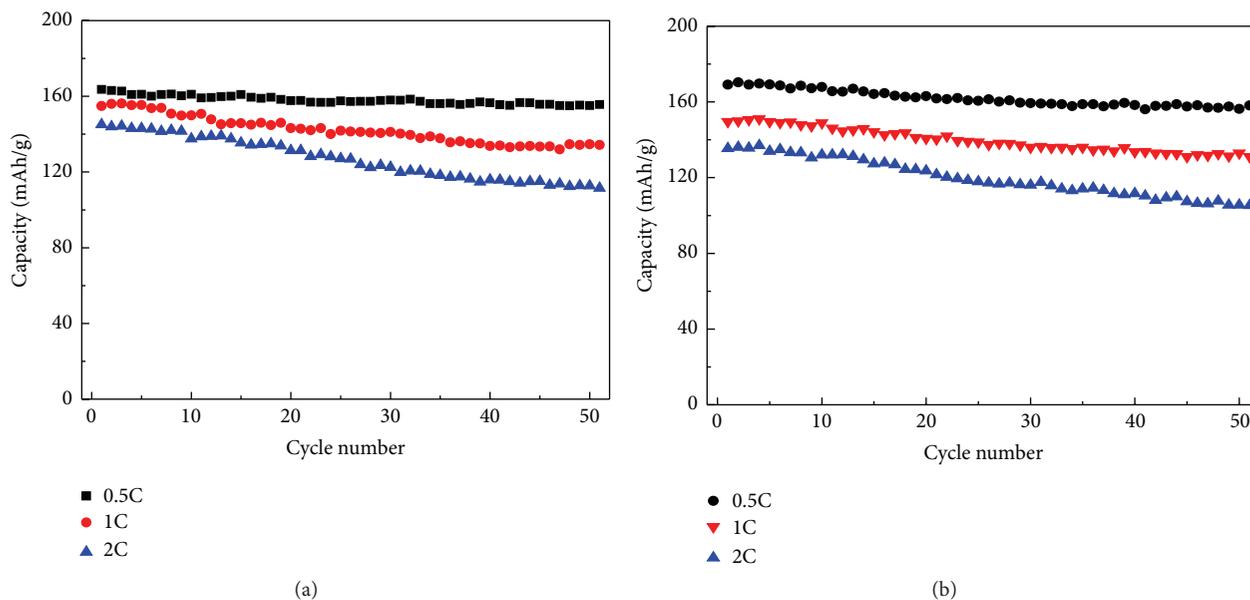


FIGURE 4: Cycling performances of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ((a) 2%; (b) 0) at different C-rate.

the 50th cycle, the electrode still obtains high discharge capacity of  $155.6 \text{ mAh}\cdot\text{g}^{-1}$ , and the capacity retention is 95.1%. Compared to  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$ ,  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  shows an obvious drop of discharge capacity from  $169.2 \text{ mAh}\cdot\text{g}^{-1}$  to  $158.1 \text{ mAh}\cdot\text{g}^{-1}$  at 0.5 C after 50 cycles; then the capacity retention is 93.4%. Furthermore, with the increase of C-rate, the cyclic performance of these two samples has a sharp decline because of the capacity irreversibility. Therefore, the cyclic performance of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  is enhanced at low C-rate. On the one hand, Zn-doping is beneficial to stabilize the structure of cathode materials and reduces the cation mixing in the electrolyte at high voltage. On the other hand, the stronger the Zn-O bond, the weaker the Li-O bond, which is beneficial to lithium ion migration [17].

Cyclic voltammogram is carried out to investigate the electrochemical performances of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  cathode materials. Figure 5 exhibits the cyclic voltammogram of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ( $x = 0, 0.02$ ) between 2.5 V and 4.5 V at a scan rate of 0.1 mV/s. As is shown in Figure 5, the curves of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$  are similar to that of pristine  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$ , without any impure peaks, which indicates that Zn-doping does not change the crystal structure of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{0.98}\text{Zn}_{0.02}\text{O}_2$ , as the XRD pattern (Figure 1) shows. Furthermore, Zn-doping leads to the potential translation, and the higher the redox potential peaks, the wider the application in the high working potential. The anodic and cathodic peaks center at around 3.882 and 3.681 V for Zn-0 and 3.941 and 3.791 V for Zn-2, respectively, and the voltage differences between the oxidation and reduction are 0.15 V for Zn-2, which is less than that (0.201 V) for Zn-0. Thus, bits of Zn-doping reduce the polarization and enhance the reversibility possibly due to improved order structure of the cathode materials.

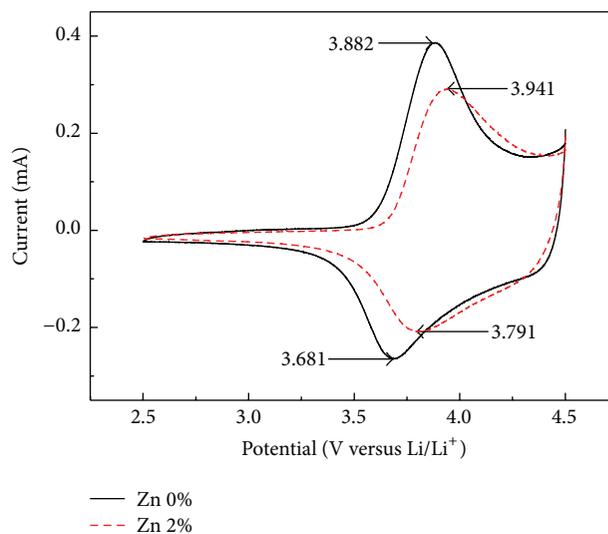


FIGURE 5: Cyclic voltammograms of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ( $x = 0, 0.02$ ) between voltages of 2.5–4.5 V at a scan rate of 0.1 mV/s.

#### 4. Conclusion

In this work,  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  ( $x = 0, 0.02, 0.05, 0.08$ ) is synthesized by doping the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$  with Zn via sol-gel route. The Zn-doping does not change the crystal structure and morphology of  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  composite; it can enhance the electrochemical performance of the  $\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})_{1-x}\text{Zn}_x\text{O}_2$  as cathode material assembled in form of lithium ion battery. With various Zn-doping content, the cathode materials can reach an initial charge/discharge capacity of 188.8/162.9 and

179.0/154.1 mAh·g<sup>-1</sup> for Zn-2 and Zn-5, and the Zn-2 has the greatest capacity retention at high voltage of 4.4 V at 0.1C. Moreover, no obvious capacity decay after 50 cycles at room temperature was observed for the Li(Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>)<sub>0.98</sub>Zn<sub>0.02</sub>O<sub>2</sub>. Reduced electrode polarization, enhanced capacity reversibility, and excellent cyclic performance make the Li(Ni<sub>1/3</sub>Co<sub>1/3</sub>Mn<sub>1/3</sub>)<sub>1-x</sub>Zn<sub>x</sub>O<sub>2</sub> be a promising lithium ion cathode material.

## Conflict of Interests

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

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