



Synthesis and Characterization of Nano SnO₂ Modification on LiNi_{0.8}Mn_{0.1}Ni_{0.1}O₂ Cathode Materials for Lithium Ion Batteries

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The precursor material of Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)₂ was prepared by a co-precipitation method to obtain the layered cathode materials LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ (811) through roasting with LiOH·H₂O. Then the SnO₂-modified samples were obtained by adding tin oxide into the ball mill. It was found by XRD characterization of the original and modified samples, that the addition of SnO₂ did not change the layered structure of the raw materials. Microstructure and distribution of the SnO₂ were analyzed with SEM and EDS. Charge/discharge tests indicated that the SnO₂-modified phases improved the cycling stability and rate capability. The results of CV test showed that the polarization of SnO₂ modified electrode was smaller. AC impedance showed that SnO₂/LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ electrode had smaller *R*_{ct} and *R*_f value. The above test results indicate that the modification of SnO₂ improves the electrochemical performance of cathode material LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂.

Keywords: Li-ion battery, Ni-based cathode materials, SnO₂ modification, electrochemical performances, lower electrode polarization

INTRODUCTION

Layered Ni-based cathode materials have attracted more and more attention due to their high energy density and environmental friendliness. Among these materials, LiNi_{0.8}Mn_{0.1}Co_{0.1}O₂ (811) material has become the focus of many pursuits because of its high specific discharge capacity, low material cost, and environmental protection concept. However, the stability and safety problems of this material are more prominent. Researchers have carried out many modification studies to solve the problems existing in LiNi_{0.8}Mn_{0.1}Co_{0.1}O₂ (811), in which doping and coating are generally recognized as effective methods. Researchers believe that doping can reduce the probability of phase transition during the cycle, improve the structural stability and contribute to electrochemical performance, doping cations such as Cr³⁺, Ti⁴⁺, Rb⁺, Al³⁺, and Mg²⁺ (Li et al., 2010, 2013; Yuan et al., 2014; Du et al., 2015; Dong et al., 2017; Zhang et al., 2017). Semiconductive or insulating materials are usually adopted to coat the LiMO₂ phase because it has the functions of protecting active substances from diffusion and dissolution in electrolyte, improving electronic conductivity, ensuring surface structure stability and avoiding corrosion of anode materials. The materials selected for coating are mainly some substances with relatively stable chemical properties, such as oxides, fluorides, salts and polymers. As previously reported, AlF₃, Al₂O₃, TiO₂, ZnO,

Li₄SiO₄, LiAlO₂, and SnO₂ (Yang et al., 2012; Lee et al., 2013; Wang et al., 2015; Dai et al., 2016; Lai et al., 2016; He et al., 2017; Srur-Lavi et al., 2017; Liua et al., 2018; Zheng et al., 2018; Xie et al., 2019) were used as effective coating materials for lithium battery cathode materials. The coated materials contribute to more stable SEI film, which can slow down the decomposition of electrolyte and protect bulk materials from corrosion during charging and discharging. For instance, SnO₂ was adopted to improve the electrochemical properties in many cathode materials with better results such as LiCoO₂ (Hudaya et al., 2014), LiFePO₄ (Ziolkowska et al., 2013), and LiMn₂O₄ (Ma et al., 2017), LiNi_{1/3}Co_{1/3}Mn_{1/3}O₂ (Luo et al., 2015), Li_{1.2}Mn_{0.54}Co_{0.13}Ni_{0.13}O₂ (Li B. et al., 2016), LiNi_{0.8}Co_{0.15}Al_{0.05}O₂ (He et al., 2017; Xie et al., 2019). However, the influence of SnO₂ modification on electrochemical properties of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ cathode materials has rarely been researched.

In this work, SnO₂/LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ was prepared by facile ball-milling post-treatment. The structure and morphology of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ with or without SnO₂ modification were investigated and the charge transfer resistance and electrochemical properties of SnO₂ modification on the LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ material were discussed in detail.

MATERIALS AND METHODS

Synthesis and Characterization

The layered Ni-rich material of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ was prepared by LiOH·H₂O and Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)₂ precursor formed by a simple continuous co-precipitation method through high temperature solid phase reaction. In this preparation process, excessive lithium hydroxide 5% (Li et al., 2012), 550°C for 6 h, 800°C for 12 h, and oxygen is introduced into the whole process. SnO₂-modified phases were further obtained by

mechanical ball milling treatment. The mass percentage of SnO₂ in composites is as follows: 1, 2, 3, and 4 wt%.

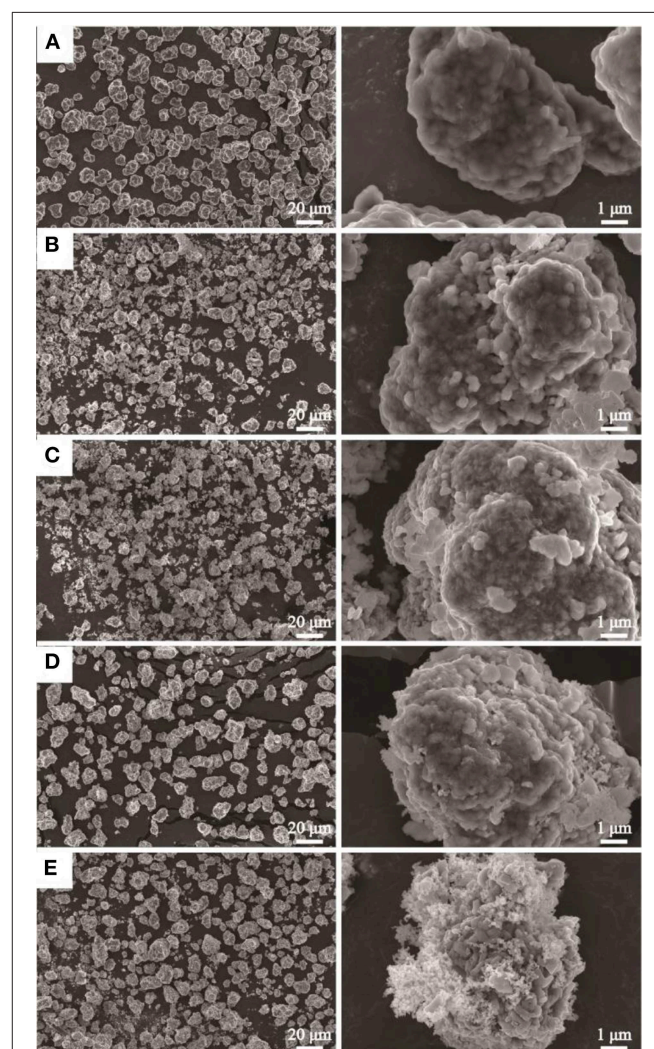
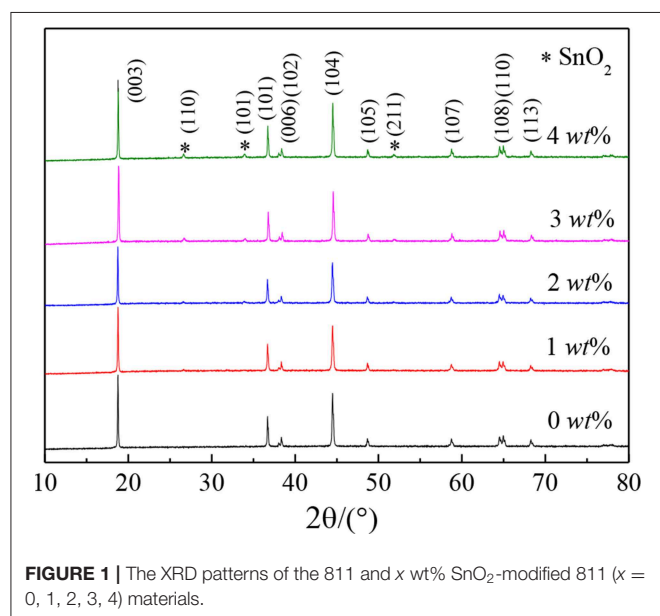
The X-ray diffraction was measured on Bruker D8 Advance X-ray Diffractometer. The morphology of the material was examined by ZEISS EVO/MA10 electron microscope. Cyclic voltammetry (CV) and EIS measurements of the cells were collected on Ivium-n-Stat electrochemical workstation.

The cathode materials were assembled into CR2032 type button batteries to detect electrochemical performances. The preparation of cathode active materials slurry and the assembly of batteries can be referred to Reference (Li et al., 2018).

RESULTS AND DISCUSSION

XRD Phase

Figure 1 shows the structure of pristine LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ (NCM) materials and its coating process with SnO₂. As shown in the figure, the cathode materials are essentially the same



and belong to a hexagonal α -NaFeO₂ structure with a space group of $R\bar{3}m$, the lattice constant are very close which means that the crystal structure of NCM is not affected by coating with nano SnO₂. Moreover, the distinct splitting of (006)/(102) and (108)/(110) indicates the products possess well-developed

layered structure (Li G. Y. et al., 2016). The XRD plots of SnO₂ modification samples also show the diffraction peak at about 27 degree representing for the plane (110) of SnO₂, which becomes obvious with the increase of the concentration of SnO₂.

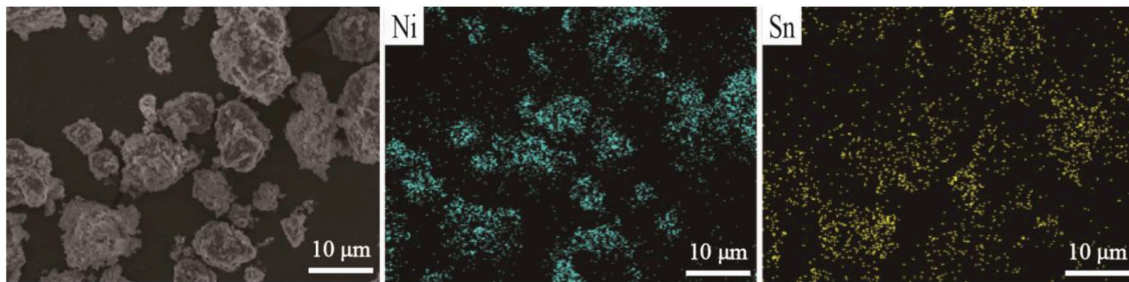


FIGURE 3 | EDS maps of the 3 wt % SnO₂-modified 811 material.

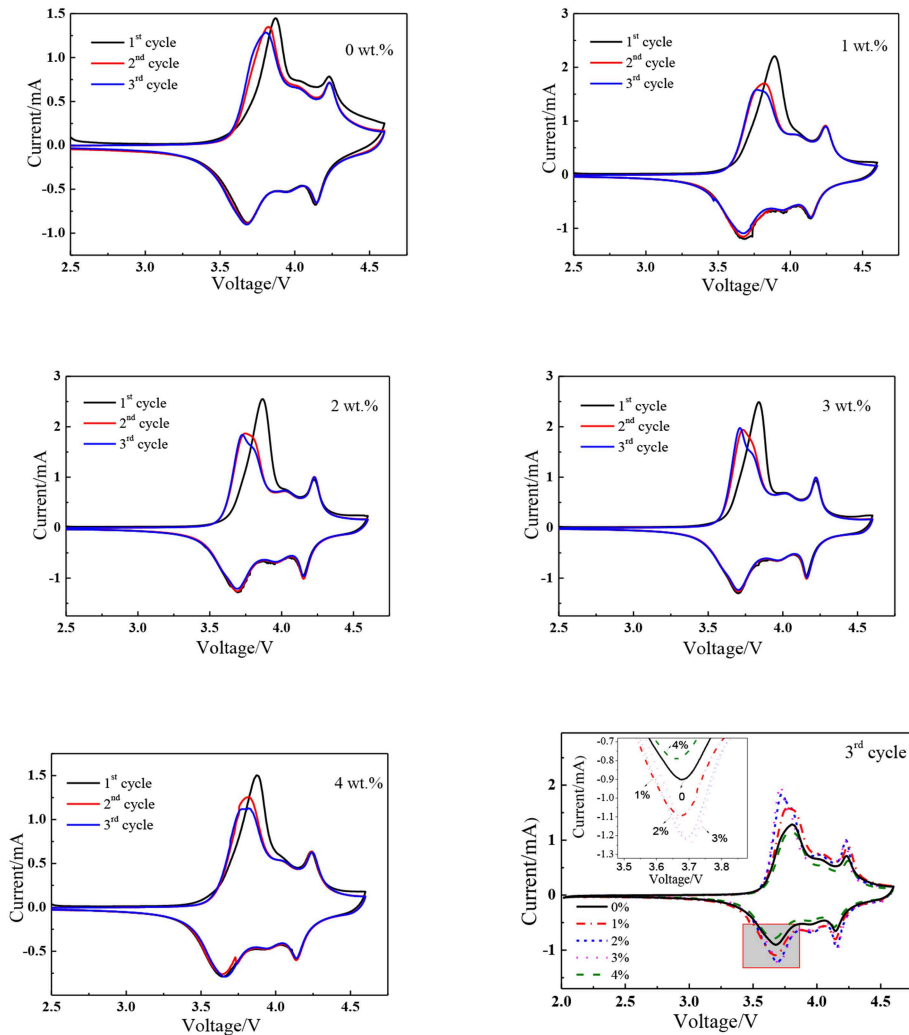


FIGURE 4 | Cyclic voltammograms of pristine and SnO₂-modified materials.

Microtopography

The SEM images of pristine and nano SnO₂-modified samples are shown in **Figure 2**. It is clearly seen that the second particles of pristine samples (the diameter is about 10 μm) are made of abundant primary nanoparticles which the surface is smooth. Compared with the pristine sample, the surface of the sample modified by SnO₂ appears rough, and some bright white spots can be seen because the nano tin dioxide particles partially cover it. However, when the mass percentage of SnO₂ is 4 wt%, the SnO₂ nanoparticles begin to aggregate and distribute unevenly on the surface of the original particles which will reduce the electro-chemical performance. The EDS spectra of 3 wt% SnO₂ modified specimen are shown in **Figure 3** which is also used to confirm the distribution of SnO₂ particles on the surface of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ cathode materials. As can be seen from the figure, the tin element covers the surface of the cathode material relatively evenly.

Electrochemical Performance

The cyclic voltammograms for the first, second, and third cycle were tested in the range of 2.5–4.6 V with a scanning rate of 0.1 mV s⁻¹ to clearly obtain the different redox behaviors of pristine and nano SnO₂-modified samples. As shown in **Figure 4**, there are three redox couples in the cyclic voltammogram curves of 2.5–4.3 V voltage range (Abraham et al., 2003; Belharouak et al., 2006), indicating the polyphase transition from hexagonal to monoclinic (H1/M), monoclinic to hexagonal (M/H2), hexagonal to hexagonal (H2/H3). The major anodic peak of the first cycle is different from the second and third cycle which may be partially attributed to the formation of solid electrolyte inter-phase (Zhong et al., 2015, 2016) or the

cation mixing (Zhong et al., 2015) in the first cycle. There is only small shift of the major anodic peak in the second and third cycle. This means that the material has good intercalation and deintercalation reversibility of lithium ions which facilitates the good electrochemical properties. As shown in **Table 1**, nano SnO₂-modified samples exhibit smaller potential difference between anodic peak and cathodic peak ($\Delta\psi/V$) compared to the pristine sample. The value of $\Delta\psi/V$ is only 20 mV when the tin oxide content is 3 wt%. It can be seen from the above data that nano SnO₂-modification can reduce electrode polarization.

Figures 5A,B are cycling performance curves of pristine and SnO₂-modified materials between the voltage of 2.8–4.35 V at 0.2C and 1C rate. The initial specific capacities of pristine and SnO₂-modified samples at 0.2C rate (1, 2, 3, and 4 wt%) were, respectively, 185.7, 181.3, 183.375, 187.5, and 179.9 mAh/g. The corresponding capacity was maintained with 85.25%, 90.40%, 91.88%, 90.77%, and 88.72 wt% after 50 cycles. The capacity of each material decreases rapidly at 1C rate, however, compare to the pristine, the capacity retention rate increased from 74.23 to 87.43% when the SnO₂ content is 3 wt%. All these results attributed to the fact that the proper addition of SnO₂ not only improves the conductivity of the material, but also reduces the

TABLE 1 | Values of CV peaks for pristine and SnO₂-modified materials in the third cycle.

SnO ₂ content (wt%)	ψ_1/V	ψ_2/V	$\Delta\psi_{12}/mV$
0	3.806	3.674	132
1	3.776	3.668	108
2	3.727	3.688	39
3	3.718	3.698	20
4	3.769	3.659	137

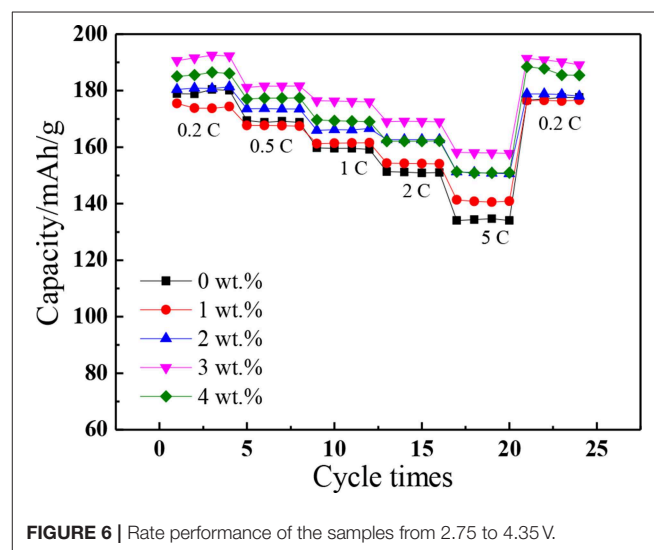


FIGURE 6 | Rate performance of the samples from 2.75 to 4.35 V.

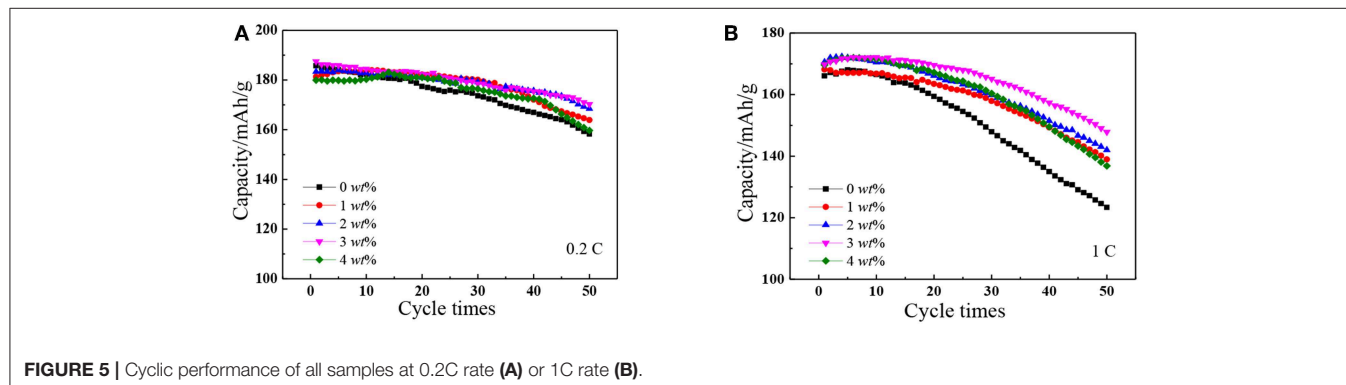


FIGURE 5 | Cyclic performance of all samples at 0.2C rate (A) or 1C rate (B).

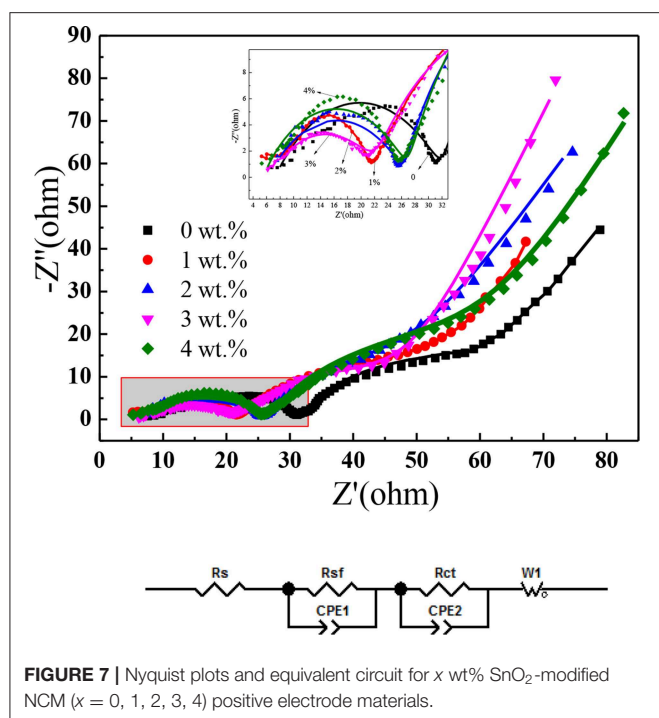


TABLE 2 | The fitting results of EIS after the 5th cycle.

x wt% SnO ₂	$R_s(\Omega)$	$R_{sf}(\Omega)$	$R_{ct}(\Omega)$
0	7.305	25.03	35.87
1	5.645	17.34	31.11
2	6.200	20.45	23.51
3	5.741	16.23	25.28
4	5.462	21.65	31.02

corrosion of the electrolyte. However, when the SnO₂ content is 4 wt%, the capacity and recyclability of cathode materials will be reduced due to the uneven distribution of SnO₂ in the surface of NCM and hinder the migration of lithium ions.

As shown in **Figure 6**, the rate performances for the pristine and SnO₂-modified materials were tested at 0.2C, 0.5C, 1C, 2C, and 5C, respectively. The average discharge specific capacity of pristine and SnO₂-NCM (the SnO₂ is 1, 2, 3, and 4 wt%) at 0.2C rate are, respectively, 179.6, 174.4, 180.8, 191.8, and 185.8 mAh/g, which, respectively, decreased to 134.3, 141.0, 150.9, 158.2, and 151.1 mAh/g when the current rate raised to 5C rate with the capacity retentions of 74.8, 68.6, 80.8, 83.5, 82.5, and 81.3%. And the discharge capacities almost restored from decay when the rate returned to 0.2C. The results showed that the existence of SnO₂ improved the rate performance of the cathode material electrode. The reason may be that the existence of SnO₂ in the electrode passivates the reaction between the cathode and the electrolyte which contributes to forming a more stable interface.

Electrochemical impedance spectroscopy measurement (EIS) technology was applied to study the internal resistance change of the five electrodes. The EIS profiles of the 5th cycled electrodes

are demonstrated in **Figure 7**. R_s , R_{sf} , and R_{ct} calculated from EIS results are tabulated in **Table 2**. All of the EIS profiles are similar and consist of two semicircles and a line, according to the reported references (Noh et al., 2013), the high-frequency semicircle represent the resistance of Li⁺ diffusion in the surface layer (R_{sf}), the mid-frequency semicircle and the low-frequency line should be, respectively, ascribed to the charge transfer resistance (R_{ct}) and the Warburg impedance of solid phase diffusion (Z_w). Compared with the original electrode, the SEI film resistance (R_{sf}) and charge transfer resistance (R_{ct}) of SnO₂ modified electrodes are slightly smaller in which the SEI film resistance of 3 wt% SnO₂ modified electrode is the smallest. Therefore, the modification of SnO₂ greatly improves the ionic conductivity, which improves the electrochemical performance of the material.

CONCLUSION

In this paper, the precursor material of Ni_{0.8}Co_{0.1}Mn_{0.1}(OH)₂ was prepared by a co-precipitation method then roasted with LiOH·H₂O to obtain the layered cathode materials LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂. SnO₂-modified phases were further obtained by mechanical ball milling treatment. The effect of SnO₂ modification on the structure, morphology and electrochemical properties of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ was mainly studied. Duo to the existence of SnO₂ in the surface of LiMO₂ active materials, the cycling stability and rate capability are greatly improved. The results of cyclic voltammetry (CV) show that the existence of SnO₂ can reduce the polarization of the electrode. When the content of SnO₂ is 3 wt%, the sample has the lowest SEI film resistance (R_{sf}) and best cycling performance performed 0.2C or 1C under the potential of 2.75–4.35 V. In conclusion, this study has potential significance for improving the cyclic and rate properties of nickel-based cathode materials.

DATA AVAILABILITY STATEMENT

The datasets generated for this study are available on request to the corresponding author.

AUTHOR CONTRIBUTIONS

CW: experimental design, data analysis and discussion, and article writing. WP: battery preparation, electrochemical performance testing, and data discussion. ZL: experimental design, electrochemical performance analysis, and discussion. YL: synthesis of cathode compounds and SnO₂ doped modification experiment. SZ: experimental design. QZ: CV performance test, results analysis, and discussion.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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