

Electrochemical properties of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ synthesized by sol-gel and co-precipitation methods

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Abstract

Layered $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ cathode materials have been prepared by sol-gel and co-precipitation methods. The structural, morphological and electrochemical properties of the materials were compared. The XRD patterns show that both the sol-gel and the co-precipitation method formed single phase materials with good layered characteristics. Electrochemical tests indicate that the material prepared by the co-precipitation method has slightly better electrochemical properties, with an initial discharge capacity of 185 mAhg^{-1} and capacity retention of 96.85 % after 50 cycles at a cycling rate of 1.0 C, as well as better capability at 7 C. The improved performances of the co-precipitation synthesized material may be attributed to the low Li/Ni disorder.

Keywords. Lithium ion batteries, Ni-rich material, co-precipitation, $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$, co-precipitation, sol-gel method.

1. INTRODUCTION

Lithium ion batteries (LIBs) have numerous outstanding features including high energy density, high conversion efficiency, no gaseous exhaust, improved safety and longer cycle life [1, 2]. The research and promotion of cathode materials are most important in the application potential of LIBs. The application of batteries utilizing layered LiCoO_2 has been limited by the relatively low specific capacity and high cost of cobalt application in plug-in hybrid vehicles (PHEVs) and all-electric vehicles (EVs) [3-5]. Recently, the layered structure series material $\text{LiNi}_{1-x-y}\text{Co}_x\text{Mn}_y\text{O}_2$ (NCM) has received increased attention [6-9]. High nickel content NCM materials, such as $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$, are very attractive cathode materials for lithium-ion batteries in electric and hybrid vehicle applications because of their relatively low cost and high reversible capacity of approximately $200 \text{ mAh}\cdot\text{g}^{-1}$ [9-11]. Various methods have been applied to synthesize Ni-rich such as solid state method [12], sol-gel method [13, 14], chloride co-precipitation [15], and co-precipitation method [16-18]. However, the high nickel content NCM materials are more difficult to synthesize in consistent quality due to the difficulty in completely oxidizing the Ni^{2+} to Ni^{3+} , even in pure O_2 atmosphere. Incomplete oxidation will eventually lead to impurities, large cation disorder and lithium deficiency. Furthermore, high nickel cathode

materials are not quite stable when exposed to air for a long time, because they can react with CO_2 or H_2O in air to form Li_2CO_3 or LiOH [10]. This process can be accelerated as the temperature is elevated [19], which implies that when calcined in air, there is also a possibility of forming some Li_2CO_3 or LiOH impurities. Co-precipitation method can be classified into two different strategies, namely carbonate co-precipitation method and hydroxide co-precipitation method. The latter one is a more efficient technology and most often used in industry which can easily provide homogeneous precursor $[\text{Ni}_x\text{Mn}_y\text{Co}_{1-x-y}](\text{OH})_2$ to get ideal homogeneous and high performance $\text{LiNi}_x\text{Mn}_y\text{Co}_{1-x-y}\text{O}_2$ cathode material with controllable morphology, high tap-density and better process ability. Co-precipitation is a commonly used method to synthesize material with high homogeneity, high tap-density and good stoichiometry.

In this work, we report on the synthesis of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ cathode materials by sol-gel (SG) and co-precipitation (CP) methods. The effects of different preparation methods on the structure, morphology and electrochemical performance of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ cathode materials are investigated.

2. METHODS

2.1. Preparation of materials

2.1.1. Sol-gel method (SG)

A mixture of LiNO_3 (98 %, Sigma-Aldrich), $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (98 %, Sigma Aldrich), $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (98 %, Sigma-Aldrich) and $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (97%, Sigma-Aldrich) with a molar ratio of $\text{Li}:\text{Ni}:\text{Co}:\text{Mn} = 1.05:0.8:0.1:0.1$ was dissolved in distilled water. Citric acid (99%, Sigma-Aldrich) was also dissolved in distilled water in a separate container (the molar ratio of metal ions: citric acid = 1:1). The two solutions were mixed together, and the pH of the solution was adjusted to 7.0 by adding $\text{NH}_3 \cdot \text{H}_2\text{O}$ (Sigma-Aldrich). The mixture was stirred for 24h at room temperature. The solution was evaporated under continuous stirring at 80 °C until the viscosity green aquogel was formed. After drying at 120 °C in a drying oven overnight, the xerogel was crushed, subsequently heated at 480 °C for 4 h in oxygen atmospheric to decompose the organic constituents and nitrate components. The sample was then grounded, pelletized and calcined at 800 °C for 16 h under oxygen atmosphere. After being cooled to room temperature, the $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ material was obtained. We denote sample name by SG-NMC

2.1.2. Co-precipitation method (CP)

The precursor $\text{Ni}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}(\text{OH})_2$ was prepared by co-precipitation method [20]. At first, stoichiometric amounts of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$, and $\text{MnSO}_4 \cdot 6\text{H}_2\text{O}$ were dissolved together in distilled water to get a transparent solution which was pumped into reactor (capacity 1.5 L) under N_2 atmosphere. At the sample time, the desired amount of NaOH solution and NH_4OH solution as a chelating agent were separately added dropwise to the transition metal sulfate solution. During the reaction process, the pH, temperature and stirring speed were carefully controlled. The spherical $\text{Ni}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}(\text{OH})_2$ powders were washed with de-ionized water and dried vacuum at 110 °C for 24 h. Finally the $\text{Ni}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}(\text{OH})_2$ precursors were mixed with 5% excess $\text{LiOH} \cdot \text{H}_2\text{O}$ and preheated at 480 °C for 5 h and at 800 for 16 h in air atmosphere to obtain the target compound of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ powders. We denote sample name by CP-NMC.

2.2 Characterizations

The crystalline structure of samples was characterized by X-ray diffraction (XRD) measurements using a Rigaku D Max/2000 PC with a CuK_α radiation in the 2θ angular range of 10 to 80°

at a scanning rate of 4° min^{-1} . The particle morphology and element composition of the powders were observed by using scanning electron microscope (SEM, Hitachi S-4800).

2.3. Electrochemical Characterization

Electrochemical cycling of the synthesized materials was performed in coin cells (CR2016) at room temperature. The cathode was prepared by tape casting a mixed slurry onto aluminum foil (battery grade) by doctor blade. The slurry was composed of 80 wt.% active cathode material, 10 wt.% Super-P carbon black, and 10 wt.% polyvinylidene difluoride (PVDF, Kynar, reagent grade) binder dissolved in N-methyl 1-2-pyrrolidone (NMP, Sigma-Aldrich, N99%). After drying the tape casted cathodes were dried overnight at 120 °C in a vacuum chamber, and the CR2016 coin cells were assembled in an argon filled glove box (moisture lower than 2 ppm). 1M LiPF_6 (Aldrich, ≥ 99.99 %) in 1:1 ethylene carbonate (EC, Sigma, 99 %)/diethyl carbonate (DEC, Aldrich, ≥ 99 %) was used as electrolyte, lithium foil as anode and Celgard 2400 membrane as separator. The cathode electrode loading was about 7 mg cm^{-2} . After assembly, the cells were allowed to rest for 15 h before electrochemical characterization. For the rate capability test, the cells were charged to 4.3 V with a current density of 0.1, 0.5, 1.0, 3.0, and 7.0 C, then kept at 4.3 V until the current density was below 0.1 C (18 mA g^{-1}), followed by discharging at the same rate as the charging rate. Long term cycling was performed at 1.0 C for 50 cycles. Cyclic voltammetry of the electrodes was obtained from test cell with a VMP3 electrochemical workstation (Bio-Logic, France) in the potential range of 3.0-4.3 V at a scanning rate of $0.1 \text{ mV} \cdot \text{s}^{-1}$.

3. RESULTS AND DISCUSSION

The morphology of the $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ powders synthesized by the two different methods, investigated by scanning electron microscopy, was shown in Fig. 1.

The powders of the CP_NMC sample consist of particles with a diameter of 100-200 nm while that is 10- 20 nm of SG_NMC sample. It seems that the sol-gel sample has smaller primary particle size but is in a serious aggregation. The aggregation makes it more difficult to break up during preparation of the electrode, which would give a smaller total surface area and hence less active surface area in contact with the electrolyte compared to the more porous

sol-gel synthesized sample.

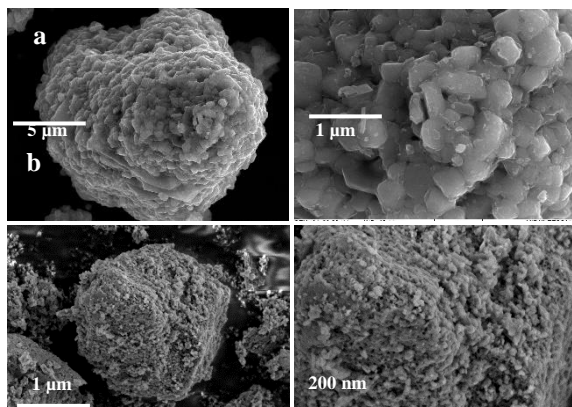


Figure 1: SEM pictures of the (a) CP_NMC and (b) SG_NMC samples

Fig. 2 displayed the X-ray diffraction patterns (XRD) of the $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ powders synthesized via sol-gel and co-precipitation method. All the diffraction peaks can be indexed on the basis of a hexagonal structure of $\alpha\text{-NaFeO}_2$ -type (space group R-3m) [21], and no impurity phase is detected in the patterns. In the layered structure, a good resolution of the (006)/(102) and the (108)/(110) reflection pairs is typical of an ideal layered structure [22]. The lattice parameters results and reliability factors are summarized in table 1. The relative intensities of the certain peaks in XRD and the value of c/a demonstrate the crystallization and the level of anti site disordering between Ni^{2+} and Li^+ . The lattice parameter a , c and ratio c/a both of two samples are nearly similar.

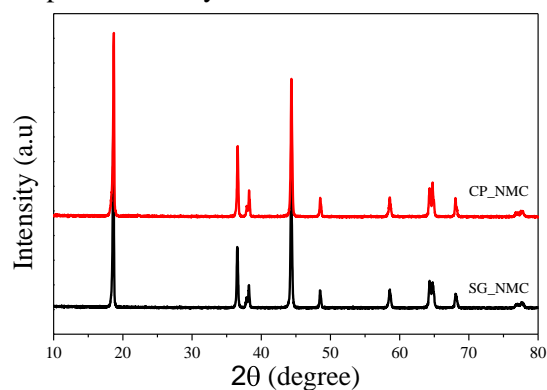


Figure 2: The XRD pattern of the SG_NMC and CP_NMC samples

Cation mixing is known to deteriorate the electrochemical performance of the layered materials. The intensity ratio of I_{003}/I_{104} is a sensitive parameter for determining the cation distribution in the lattice of the layered oxide, and a value lower than 1.2 indicates a high degree of cation mixing

which is an indication of undesirable cation mixing. From data in table 1, R_w of two samples is greater than 1.2 which shows low Ni^{2+} and Li^+ antisite disordering. It can be provided high electrochemical performance.

Table 1: Lattice parameters of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ prepared with sol-gel and co-precipitation method

LNMCO	a	c	c/a	Rw
SG-NMC	2.8378	14.2019	5.004	1.4018
CP-NMC	2.8753	14.2287	4.9486	1.4131

The electrochemical performances of $\text{Li}/\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ cells have been investigated. The initial charge - discharge curves of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ material at a discharge rate of 0.1C (18 mA g^{-1}) in the potential range from 3.0 to 4.3 V at 25°C were shown in Fig.3 (a). The material exhibited one plateau during the first charge, due to the existence of one lithium de-insertion process. This plateau is associated to the delithiation that corresponds to the oxidation of $\text{Ni}^{2+} \rightarrow \text{Ni}^{4+}$. The initial discharge capacity of the sample synthesized by the co-precipitation method is 185 mAhg^{-1} with a columbic efficiency of about 84.09 %. The result is comparable to that of the $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ reported in literature [12], which was also prepared by co-precipitation method. The sample synthesized by the sol-gel method delivered an initial discharge capacity of 182 mAhg^{-1} with a columbic efficiency of about 86.25 %.

Fig. 3 (b) showed the cycling performance of $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ synthesized by the sol-gel and the co-precipitation methods cycled at a current rate of 1 C between 3.0 and 4.3 V. The CP_NMC sample showed the highest specific discharge capacity. The cell delivers a capacity of 165.232 mAhg^{-1} at 1st cycle and 160.035 mAhg^{-1} at 50th cycle, while capacity of 161.395 mAhg^{-1} at 1st cycle and 131.729 mAhg^{-1} at 50th cycle of SG_NMC sample, respectively. The samples synthesized by the sol-gel method show capacity retention of 87.83 % and 81.61 % for 30 and 50 cycles, respectively.

The sample synthesized by co-precipitation shows higher capacity retention of 98.15 %, and 96.85% during the same cycling period. From the results of initial charge-discharge, rate capability and cycling performance, we found that the sample synthesized by the co-precipitation method displayed slightly higher initial discharge capacity, improved capacity retention and better rate capability at high discharge rates compared to the sample synthesized by the sol-gel method. The

better electrochemical performance of the co-precipitation synthesized material might be attributed to the lower Li/Ni disorder, the latter providing better contact between the electrolyte and the active material. The fact that the sol-gel sample has a higher Li/Ni disorder leads to lower Li diffusivity, which will drive down its capacity at high rates.

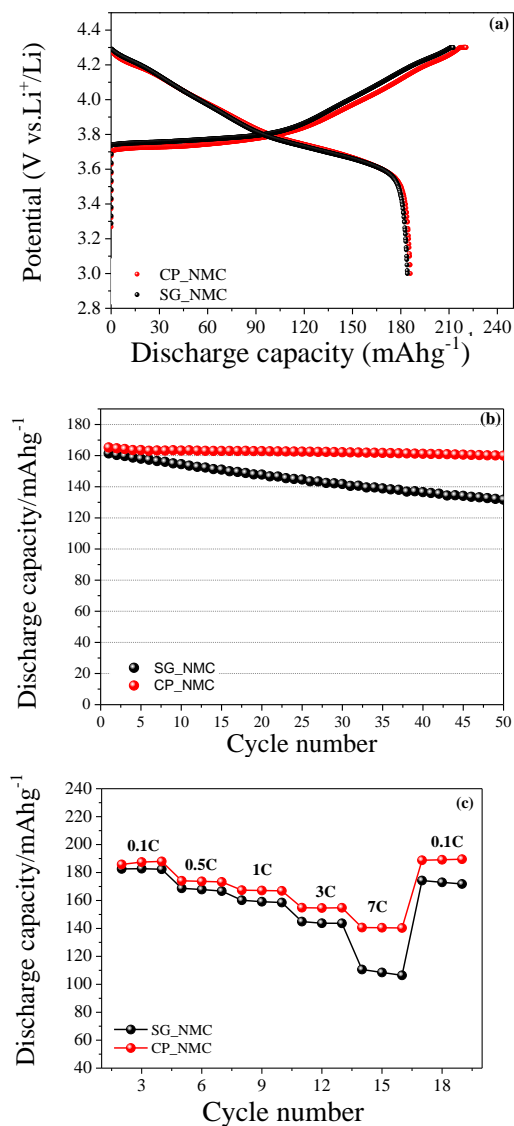


Figure 3: Electrochemical properties of samples in a voltage window of 3.0-4.3 V at 25 °C: (a) Initial charge – discharge capacity at a rate of 0.1C and (b) discharge capacity vs cycle number at a rate of 1C and (c) rate capability at various C-rates

Table 2: Specific discharge capacity at various C-rates of SG_NMC and CP_NMC samples

LNMCO	0.1C	0.5C	1C	3C	7C
SG-NMC	182.59	168.72	160.17	144.88	110.60
CP-NMC	185.79	174.12	167.36	154.85	140.62

Fig.3 (c) showed the rate performance of the samples with various current densities between potential limits of 3.0-4.3 V. The cell is charged at a current density of 92.5 mA_g⁻¹ (0.5C) before each discharge test. The cells are first cycled at 0.1 C (18.5 mA_g⁻¹) and then at 0.5 C (92.5 mA_g⁻¹), 1 C (185 mA_g⁻¹), 3 C (555.5 mA_g⁻¹) and 7 C (1285 mA_g⁻¹) for every discharge cycles. From 0.1C to 1C, the two materials show similar electrochemical performances. However, when the discharge current rate is increased to 7 C, the sample synthesized by the co-precipitation method shows more stable and higher capacity compared to the sample synthesized by the sol-gel method which was shown more detail in Table 2. For instance, the discharge capacity of the CP_NMC sample is about 167 and 140 mAh_g⁻¹ at 1 C and 7 C, while the discharge capacity is 160 and 110 mAh_g⁻¹ of SG_NMC sample, respectively. When the discharge current rate goes back to 0.1 C, the discharge capacity of the two samples showed much difference, the capacity of CP_NMC sample returned to approximately 188 mAh_g⁻¹ higher than that of SG_NMC sample. The difference in the discharge capacity between the sol-gel and the co-precipitated samples at high C-rates can possibly be explained by the faster charge transfer in the co-precipitation synthesized sample due to higher density and higher porosity.

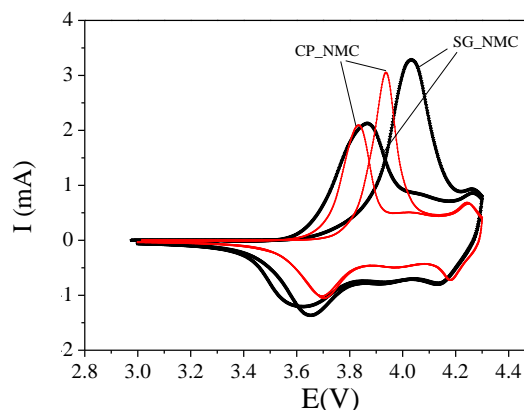


Figure 4: Cyclic voltammetry of 2 samples SG_NMC and CP_NMC at 0.1 mV s⁻¹ in a voltage range 3.0-4.3 V

Fig. 4 shows the CV curves of LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ synthesized via the sol-gel and the co-precipitation method for the initial two cycles in the potential range of 3.0-4.3 V at a scanning rate of 0.1 mVs⁻¹. During charge-discharge, layered cathode materials with high nickel content such as LiNiO₂ and LiNi_{0.8}Co_{0.15}Al_{0.05}O₂, often exhibit four different phases (one monoclinic phase, M, and three hexagonal phases, H1, H2, and H3) [23].

Table 3: Redox peak of cyclic voltammetry analysis of CP_NMC and SG_NMC electrodes

Sample	Peak	Oxidation (V)	Reduction (V)	Polarization (V)
SG_NMC	1 st cycle	4.032	3.653	0.379
	2 nd cycle	3.866	3.611	0.255
CP_NMC	1 st cycle	3.936	3.703	0.233
	2 nd cycle	3.831	3.699	0.132

As shown in Fig. 4, the curves of the two samples both show characteristic of layered oxide cathodes with a couple of significant redox peaks corresponding to Ni²⁺/Ni⁴⁺ reaction. However, the activation peak disappears in the second cycle and a broad peak appears at about 3.85 V, which is the main anodic peak for the newly formed oxide, containing the oxidation of Ni and Co element. The Ni²⁺/Ni⁴⁺ reaction contributes mainly to the charge/discharge capacity of NMC and the difference of their peak voltages (ΔE_p) reflects the polarization for lithium insertion or extraction. Through comparing the oxidation peaks of the SG_NMC and CP_NMC samples (table 3), it is clear that the polarization of CP_NMC sample is smaller than that of SG_NMC, implying that the kinetics behavior has been improved. The improvement herein can be ascribed to the CP_NMC which is good for the extraction of lithium during charge. It helps to understand why the material is often facing rapid capacity loss during cycling as shown in Fig. 3(b).

4. CONCLUSION

In conclusion, we reported high capacity nickel-based LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ cathode materials synthesized by sol-gel and co-precipitation methods. The effects of different synthesis methods on the crystal structure, morphology, and electrochemical properties of the materials have been investigated. The two samples prepared by the different methods show good layered characteristics and high crystallinity. The electrochemical study showed that the sample prepared by the co-precipitation method has better electrochemical properties, with a higher initial discharge capacity of 185 mAhg⁻¹ and capacity retention of 96.85 % after 50 cycles at a cycling rate of 1.0 C, as well as better capability at 7 C. The better electrochemical properties of the co-

precipitation sample may be ascribed to the lower cation (Li/Ni) disorder.

The LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ material is attractive as a positive electrode material for high power energy lithium-ion batteries, and this work shows that low cost and environmentally friendly co-precipitation synthesis is a very promising method to achieve high performance cathodes.

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