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International Journal of Ionics The Science and Technology of Ionic Motion

ISSN 0947-7047

lonics DOI 10.1007/s11581-018-2800-3





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ORIGINAL PAPER



Synthesis of Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ cathode materials for lithium-ion rechargeable battery by glycine-nitrate combustion process

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Received: 28 June 2018 / Revised: 9 October 2018 / Accepted: 12 November 2018 © Springer-Verlag GmbH Germany, part of Springer Nature 2018

Abstract

This study was based on developing Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ (x = 0.04, 0.08, 0.11, 0.22, and 0.33) materials by substituting expensive Co with Ba, for the use in the cathode of rechargeable lithium-ion batteries (LIBs). Glycine-nitrate combustion method, which is a low-cost combustion technique, was employed to synthesize spherical shaped micron size secondary particles formed by densely agglomerated primary particles. The phase analysis performed by the X-ray diffractometry revealed the formation of the required layered phase of R-3m structure with trace amounts of a secondary phase. Furthermore, these Basubstituted novel materials showed considerably higher electrical conductivity than those of the Li(Ni_{1/3}Co_{1/3}Mn_{1/3})O₂ base material. In the cell performance studies, the Ba-substituted cathode materials synthesized in this study showed slightly lower initial discharge capacity of 162.4 mA h g⁻¹ but with considerably improved cycle performance compared to those of the Li(Ni_{1/3}Mn_{1/3})O₂ base material (187.7 mA h g⁻¹). More importantly, the Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 material clearly showed its ability to eliminate and prevent structural transformation usually associated with excess Li extraction at potentials above 4.5 V. Therefore, the Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 material can be proposed as a potential candidate for the high-voltage cathode application of LIB.

Keywords Li-ion batteries · Doping · Materials preparations · Cathodes

Introduction

Lithium-ion batteries (LIBs) are emerging as promising potential candidates in portable power sector in view of their wide range of applications from portable consumer electronics to electric vehicles. The LIB has been usually involved the use of lithiated transition metal oxides, namely LiCoO₂, LiMn₂O₄, and LiFePO₄. Moreover, Li(Ni_{1/3}Co_{1/3}Mn_{1/3})O₂ has been extensively investigated due to its advantages such as high discharge capacity, low cost, and environmental

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benignity compared to conventional commercial cathode materials such as $LiCoO_2$ [8, 11]. However, several issues such as low first coulombic efficiency, the poor cycling stability, and rate performance at high voltage are obstructing its application as a cathode material in LIBs [9, 17]. Recently, substitution of alkali metals and transition metals for Co has been shown to improve the electrochemical property of this $Li(Ni_{1/3}Co_{1/3}Mn_{1/3})O_2$ important material system [10].

Previous studies have indicated that the presence of Ba cations in the inter-slab is expected to provide structural stability when the layered transition metal oxides are cycled to 4.5 V [12–14]. However, studies on Ba substitution into these three component layered transition metal oxides of Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ have not yet been investigated. Therefore, this study addressed it by partially substituting Ba for Co in the Li(Ni_{1/3}Mn_{1/3}Co_{1/3}O₂ system. It was carried out by synthesizing x = 0.04, 0.08, 0.11, 0.22, and 0.33 compositions by employing the glycine-nitrate combustion (GNC) method and calcining at 900 °C, with the aim of achieving improved electrochemical performance to them.

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Powder synthesis by GNC method

For this study, $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$ powders were synthesized by the glycine-nitrate combustion (GNC) method. For that, stoichiometric amounts of metal nitrates, LiNO₃, $Ni(NO_3)_2 \cdot 6H_2O$, $Co(NO_3)_2 \cdot 6H_2O$, and $Mn(NO_3)_2 \cdot 4H_2O$, Ba(NO₃)₂ (Merck, Germany) were mixed with distilled water. Glycine was added as the fuel to the nitrates while keeping glycine to nitrate (G:N) ratio at 0.6 ([1, 2]). The precursor solution was thoroughly mixed using a magnetic stirrer hotplate at room temperature for 2 h, and then at 60 and 100 °C for 1 h each. After that, the temperature of the precursor was increased in steps of 25 up to 300 °C. During this process, the resultant viscous liquid got ignited and underwent self-sustaining combustion producing an ash composed of the oxides. Finally, the ash product was further calcinated at 900 °C in air for 2 h in a muffle furnace to obtain the final oxide product.

Powder characterization

X-ray diffractometry (XRD, Siemens D5000) using CuK α radiation was used for phase analyze while scanning electron microscopy (SEM, FEI Quanta 200 FEG-ESEM) was employed to study the morphology and the particle size of the synthesized powders. Thermogravimetric analyses (TGA) were carried out using a Perkin-Elmer TGS-2 Thermogravimetric System. For that, calcinated powders (0.5 mg) were heated up to 800 °C in a platinum pan at a heating rate of 5 °C min⁻¹, under nitrogen atmosphere.

Powder densification and electrical characterization

Green pellets of 12 mm in diameter and 0.5 mm in length were prepared by pressing uni-axially at 250 Mpa [2]. The green pellets were subsequently sintered at 1000 °C for 2 h in the air. For the electrical conductivity measurements, the flat end surfaces of the sintered pellets were gold pasted (G3535, Agar Scientific Ltd., England) and heat treated at 750 °C in order to improve the contact between the specimen pellets and the sample holder. The d.c. four-probe electrical conductivity measurements were performed on the sintered pellets on heating and cooling in the temperature range between room temperature (25 and 200 °C), by keeping the specimens in a sample holder inside a pot furnace.

Electrochemical characterization

For electrochemical characterizations, electrodes were prepared using the slurry containing 85 wt.% of the prepared Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ active material with 5 wt.% carbon black (Sigma Aldrich) and 10 wt.% PvDF (KynarFlex 2801) in Acetone (Sigma Aldrich). The prepared slurry was then manually tape cast onto an aluminum foil by the doctor blade method. After drying, circular disk electrodes were cut from the fabricated tapes.

Coin cells (Type CR 2032) were assembled in an argonfilled glove box (< 1 ppm O₂ and H₂O) with the developed cathode electrodes, Li-foil (Sigma Aldrich) as the reference and counter electrodes, respectively. Cellgard 2400 was used as the separator with the non-aqueous electrolyte of 1 M LiPF₆ in ethylene carbonate (EC)/diethyl carbonate (DEC)/dimethyl carbonate (DMC) (1:1:1 wt%) (Selectilyte LP71, BASF, USA). Finally, the charge-discharge study of the assembled cells was carried out at C/5 rate with a cut-off voltage of 2.5– 4.6 V at 25 °C, using Scribner 580 Battery Testing System controlled by BCycle 2.0 program.

Results and discussion

Phase analysis

The X-ray diffractograms obtained on Li(Ni_{1/3}Mn_{1/3}Co_{1/3-} $_xBa_x$)O₂, x = 0.00, 0.04, 0.08, and 0.11 materials prepared in this study by calcining at 900 °C are shown in Fig. 1. Formation of phase pure hexagonal structure of α -NaFeO₂ (space group R-3m) was reported in Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ at a low level of substitution [11, 14]. However, trace amounts of a secondary phase could be identified in the X-ray diffractogram of Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 material prepared in the present study. The major diffraction peaks related to α -NaFeO₂ hexagonal structure appear to diminish with the increase of Ba addition form x = 0.04 to x = 0.33 in the present study. Similarly, clear splitting of the (006)/(102)and (108)/(110) diffraction lines had also diminished with the increase of Ba content. Moreover, a number of new emerging secondary phases can be observed with the increase of Ba content from x = 0.08 to x = 0.33. This suggests that the cations are not homogeneously distributed over the structure of these compositions by occupying alternative layers in the octahedral sites [5, 16]. The size of the Ba^{+2} (1.34 Å) ion is significantly larger than Li⁺ (0.76 Å), Ni⁺³ (0. 69 Å), Mn⁺⁴ (0.53 Å), and Co⁺³(0.54 Å); therefore, Ba could have a lower tendency to substitute for those smaller cations. This might be the reason for the presence of trace level of secondary phases even at low Ba substitution (x = 0.04) in the present study.

Particle properties

The scanning electron micrographs and energy dispersive spectra obtained on Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 material are shown in Fig. 2. According to the SEM image, the Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ powders consist of sub-micron

Fig. 1 XRD patterns of the $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2, x = 0.00, 0.04, 0.08, 0.11, 0.22, and 0.33 materials$



level secondary particles. These secondary particles further consist of densely agglomerated primary particles of a broader particle size distribution. Two types of primary particles can be clearly identified in these secondary particles. The larger particles (0.3 to 1 μ m) are rather hexagonal in shape having sharp edges with smooth and clear surfaces. In contrast, the smaller particles, which are in the range of 50–100 nm, are well rounded in shape.

EDX spectra of the synthesized Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 (Fig. 2b) show the presence of only Ni, Co, Mn, Ba, and O in the calcined powder. The atomic ratio of Na:Mn:Co:Ba in the Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04 material was determined to be approximately 9.44:10.62:9.02:0.12.

Figure 3 shows the thermal gravimetric analysis (TGA) curves and the differential thermogravimetric plots (dW/dT plots) obtained on Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.00, 0.04, 0.08, 0.11, 0.22, and 0.33 materials. The measurements were performed between 25 and 800 °C with a heating rate of 5 °C min⁻¹, under nitrogen atmosphere. x =0.04, 0.08, 0.11, and 0.22 materials show a quite a similar behavior but x = 0.33 material shows the existence of several exothermic peaks. The first plateau of weight loss observed between 100 and 200 °C is very likely related to the loss of water molecules [8]. In contrast, two prominent exothermic peaks are present in this x = 0.33 material at a temperature below 220 °C. In addition, this material consists of an exothermic peak at 440 °C and three other exothermic peaks at 524, 650, and 746 °C. The existence of such several exothermic peaks in the TGA curve of the x = 0.33 material could be related to the decomposition of the secondary phases present, as evident from the XRD analysis.

Electrical characterization

Figure 4 shows the temperature dependence of conductivity of $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2, x = 0.00, 0.04, 0.08, 0.11, 0.22,$ and 0.33 materials measured by the d.c. four-probe technique. The apparent exponential behavior, in the increase of the conductivity with the temperature of these synthesized materials, clearly indicates their semiconducting nature [10, 11]. The electrical conductivity of x = 0.08 and 0.11 materials shows $1.06 \times$ 10^{-3} S cm⁻¹ and 1.70×10^{-3} S cm⁻¹, respectively at 25 °C. So, it is significantly higher, with a factor of 10^4 , in these x = 0.08and 0.11 materials with compared to those of $Li(Mn_{1/3}Ni_{1/3}Co_{1/3})O_2$ base material [1]. The existence of some secondary phases, observed in the XRD analysis, may be the reason for this higher conductivity. The electrical conductivity of x = 0.22 and 0.33 is 3.11×10^{-06} S cm⁻¹ and 1.00×10^{-07} S cm⁻¹, respectively. It indicates a significant decrease of the electrical conductivity with respect to a very high level of Ba substitution. It may have resulted from the combined effect of diminishing phase pure hexagonal structure and increase of the secondary phases with the increase of Ba amount form x = 0.08 to x = 0.33, as evident from the XRD analysis. However, the x = 0.04 material, which is having phase pure structure, still shows a conductivity $(1.20 \times 10^{-05} \text{ S cm}^{-1})$ adequate for the LIB cathode application. In this x = 0.04 material, the electrical conductivity has increased by a factor of 10^2 compared to that of Li(Mn_{1/3}Ni_{1/3}Co_{1/3})O₂ [1].

Electrochemical characterization

For this study, CR2032 haft-cells were assembled with electrodes fabricated from the synthesized $Li(Ni_{1/3}Mn_{1/3}Co_{1/3}.$



(a)



Fig. 2 a SEM image and b EDX spectra obtained on the $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$, x = 0.04 material

 $_xBa_x)O_2$, x = 0.00, 0.04, and 0.08 materials as the working electrode together with Li foils as the counter and reference electrodes. The galvanostatic cycling was carried out between 2.5 and 4.6 V, at 0.2 C rate (1C = 140 mA g^{-1}) at 25 °C. The charge-discharge curves obtained on the half cells assembled with $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$, x = 0.00, 0.04, and 0.08 are shown in Fig. 5. All these cells exhibited charge/discharge behavior with a voltage profile similar to that reported for phase pure hexagonal structure of Li(Mn_{1/3}Ni_{1/3}Co_{1/3})O₂ reported in the literature [11, 13]. Further, it can clearly be seen that the discharge capacity of the electrode material decreases monotonically with the increase of Ba content. The initial discharge capacities of Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04and 0.08 were 162.4 and 159.3 mA h g^{-1} , respectively. They are slightly lower than those reported for the base material, $Li(Mn_{1/3}Ni_{1/3}Co_{1/3})O_2$ (187.7 mA h g⁻¹) [1, 2].

In order to identify the electrochemical redox reaction which might occur during the charge and discharge process, the respective differential capacity (dQ/dV) values were calculated from the numerical data presented in Fig. 5, Accordingly, the corresponding dQ/dV vs. V plots of x = 0.04 and 0.08 materials are presented in Fig. 6a and b, respectively. In both materials, the differential capacity plots, given in Fig. 6, show the existence of one oxidation peak and one reduction peak during the first cycle. A sharp oxidation peak can be observed at 3.78 V during the charging process, from the first to the tenth consequent cycles. In x = 0.08 material, this peak has shifted to a higher potential from 3.82 V. During the discharging process, the corresponding reduction peak can be observed at 3.74 V from first to the tenth consequent cycles. This implies the reversibility of the redox reaction associated with the lithium intercalation and de-intercalation together with the reducing capacity fading of the material during cycling [3, 6]. Moreover, an absence of oxidation or reduction peaks below 3.5 V indicates electrochemical inactivity of the Mn



Fig. 3 a TGA curves and **b** differential thermogravimetric (dW/dT) plots of Li(Ni_{1/3}Mn_{1/3}Co_{1/3x}Ba_x) O₂, x = 0.00, 0.04, 0.08, 0.11, 0.22, and 0.33 materials. The measurements were taken from 25 to 800 °C, at a heating rate of 5 °C min⁻¹, under nitrogen atmosphere

ions [6]. It is clearly evident that a second irreversible oxidation peak at potentials above 4.5 V, which is a drawback found in the base material of $Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$,



Fig. 4 The temperature dependence of the electrical conductivity of $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$, x = 0.00, 0.04, 0.08, 0.11, 0.22, and 0.33 materials



Fig. 5 Initial charge-discharge curves of half cells assembled with $Li(Ni_{1/3}Mn_{1/3}Co_{1/3}Ba_x)O_2$, x = 0.00, 0.04, and 0.08 cathode at C/5 rate $(1C = 140 \text{mAhg}^{-1})$ with a cut-off voltage of 2.5–4.6 V, at room temperature

is absent in these Ba-substituted materials [1]. The irreversible oxidation process occurring during the charging at potentials above 4.5 could be correlated to the loss of oxygen with excess Li extraction from the layered structure. This process has been completed during the first charge [15]. Therefore, reduction of irreversible capacity loss due to Ba addition, as discussed above, may be associated with the absence of the oxidation peak at potentials above 4.5 V during the first cycle [4].

Figure 7 shows the variation on discharge capacity vs. cycle number of half cells assembled with x = 0.00, 0.04, and 0.08 cathodes between 2.5 and 4.6 V. The behavior resulting from the base materials Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ (x = 0.00) under the same experimental condition is also presented in Fig. 7 [1]. The initial discharge capacity of x = 0.04 was 162.4 mA h g⁻¹. Its discharge capacity has slightly faded to 147.9 mA h g^{-1} at the 10th cycle. Therefore, it shows a slightly improved cycle performance of x = 0.04 compared to that of the base material, $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$ (x = 0.00) [1]. Interestingly, the x = 0.08 material prepared in the present study shows an improved discharge capacity retention compared to that of the base material, $Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ [1]. During the first ten cycles, the discharge capacity of x = 0.08 has faded from 145 mA h g^{-1} to 134 mA h g^{-1} . Therefore, this study revealed the possibility of increasing the cycle performance of $Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ material system with the introduction of Ba.

This enhancement of cycle performance due to the Ba addition in Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ has not been clearly identified yet. In this study, the thermogravimetric analysis shows the enhanced thermal stability by adding Ba to Li(Ni_{1/3}Mn_{1/3}Co_{1/3}). Similarly, the observed enhancement of electrical conductivity with the addition of Ba might also support this improvement in the electrochemical performance, as



Fig. 6 Differential capacity versus voltage of half cells assembled with $Li(Ni_{1/3}Mn_{1/3}Co_{1/3}xBa_x)O_2$ **a** x = 0.04 and **b** 0.08 for the first, second, and tenth cycles, at the rate of 0.2 C, between 2.5 and 4.6 V

this could decrease the charge transfer resistance of the electrode [7]. More importantly, the differential capacity plots of Ba-substituted Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂, x = 0.04, 0.08 materials prepared in this study clearly indicate the elimination of irreversible oxidation process, corresponding to the loss of oxygen at higher potentials (>4.5 V). Therefore, these Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ cathode materials, especially with x = 0.04 and 0.08, investigated in the present study can be proposed as good candidates for the rechargeable lithium-ion batteries operating under high voltages.

Conclusion

Ba-added Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂ materials were synthesized by glycine-nitrate combustion process, under a



Fig.7 Discharge capacities vs. cycle number of half cells assembled with $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$, x = 0.00, 0.04, and 0.08 cathodes, at C/5 rate (1C = 140 mAh g⁻¹) with a cut-off voltage of 2.5–4.6 V, at room temperature

shorter calcination time of 2 h. The phase analysis indicated that only lower levels of Ba additions, such as x = 0.04, could results in phase pure Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O₂. The synthesized powers show the existence of rather spherical shaped micron size secondary particles formed by densely agglomerated primary particles having a border particle size distribution. Further, the electrical conductivity study indicated a semiconducting behavior to these synthesized materials. Ba addition could significantly increase the electrical conductivity, and the electrical conductivity of the x = 0.4 material showed an increase by a factor of 10^4 compared to that of Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ base material.

The lithium-ion half cells prepared with $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$, x = 0.04 and 0.08 materials showed an initial discharge capacity of 162 and 145 mA h g^{-1} , respectively at cut-off voltage of 2.5–4.6 V. Thought these Ba-doped cathode materials had slightly lower initial discharge capacity, they showed considerably improved cycle performance compared to the those of the base material. The enhanced thermal stability and higher electrical conductivity with the introduction of Ba could be the reason for this improved cycle performance. More importantly, the Ba-added material with x = 0.04 prepared in this study showed its ability to eliminate structural transformation under excess Li extraction at potentials above 4.5 V. Therefore, these $Li(Ni_{1/3}Mn_{1/3}Co_{1/3-x}Ba_x)O_2$ cathode materials can be proposed as good candidates for the rechargeable lithium-ion batteries operating under high voltages.

Funding information This study received financial assistance from the Human Resources Development (HRD) program of the Higher Education for Twenty-First Century (HETC) project of Ministry of Higher Education, Sri Lanka.

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