SYNTHESIS AND CHARACTERIZATION OF P2-$Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ MATERIAL FOR CATHODE IN SODIUM-ION BATTERIES

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ABSTRACT

In this work, a $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ material with a P2-type structure was successfully synthesized using the simple solid state technique. The crystal structure and morphological characteristics of $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ material were comprehensively analyzed by X ray analysis, scanning electron microscopy, and energy dispersive spectroscopy. The $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ compound was used to make CR2032-type coin cells for evaluating its electrochemical characteristics. Electrochemical testing revealed that the initial charge and discharge capacities of the $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ cathode, at a current density of 10 $mA.g^{-1}$ within the voltage range of 1.5 - 4.5 V, were 127.29 $mAh.g^{-1}$ and 114.11 $mAh.g^{-1}$, respectively. After 50 charge-discharge cycles, the retained capacity is approximately 57.2% of the first cycle discharge capacity. The results show that the $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ oxide is a potential cathode for sodium ion battery applications.

TỔNG HỢP VÀ TÍNH CHẤT ĐẶC TRƯNG CỦA VẬT LIỆU P2-$Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ ỨNG DỤNG LÀM CATỐT CHO PIN NATRI-ION

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TỪ KHÓA

Pin natri-ion
Vật liệu catốt
Phản ứng ở trạng thái rắn
Vật liệu oxit gốc natri mangan
Vật liệu cấu trúc lớp

TÓM TẮT

Trong bài viết này, vật liệu $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ có cấu trúc kiểu P2 đã được tổng hợp thành công bằng phương pháp phản ứng trạng thái rắn. Đặc điểm cấu trúc và hình thái học của vật liệu $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ đã được phân tích toàn diện bằng phương pháp nhiễu xạ tia X, kính hiển vi điện tử quét và phổ tán sắc năng lượng. Vật liệu $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ đã được sử dụng để chế tạo pin đồng xu loại CR2032 và sau đó chúng được đánh giá về các đặc tính điện hóa. Kết quả cho thấy dung lượng sạc và xả ở chu kỳ đầu của vật liệu catốt $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ ở mật độ dòng điện 10 $mA.g^{-1}$ trong dải điện áp từ 1,5 V đến 4,5 V, lần lượt là 127,29 $mAh.g^{-1}$ và 114,11 $mAh.g^{-1}$. Dung lượng vẫn duy trì được 57,2% dung lượng xả của chu kỳ đầu tiên. Kết quả này cho thấy vật liệu NLMN là một catốt có triển vọng đầy hứa hẹn để ứng dụng cho pin natri-ion.

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1. Introduction

Lithium-ion batteries (LIBs) are widely used for electric vehicles and portable electronic devices. They have many favorable benefits, for instance light weight, low redox potential ($E_{Li+/Li}$ = -3.05 V) in comparison to the common hydrogen electrode, Li^+ ion with a relatively small radius, prolonged cycle durability, and excellent energy efficiency. However, lithium scarcity is becoming a critical issue as demand for batteries continues to surge, especially with the rise of electric vehicles and renewable energy storage. The increasing cost of LIBs due to the increasingly depleted supply of lithium. Researchers are actively exploring alternative batteries, such as sodium-ion, potassium-ion, and even solid-state batteries [1] - [4].

Recently, sodium-ion batteries (NIBs) have emerged as a viable and cost-effective substitute for lithium-ion batteries offering several notable advantages such as: (i) the high natural abundance and cost effectiveness of sodium resources, (ii) low redox- potential ($E_{Na+/Na}$ = -2.71 V) which is favorable for achieving high cell voltage, non toxic nature, and (iv) facile fabrication [5]. The operating principle of NIB and LIB is familiar. However, the larger ionic size of sodium makes the transportation of sodium ion in the cathode material more difficult. Therefore, it is essential to find electrode materials with a favorable structural framework for sodium ions to transport. Many materials have been found to be suitable for cathode of sodium ion batteries for instance: phosphates polyanions, AMO_2, $AMPO_4$ (in which A and M represent for alkali metals and transition metals, respectively) [6].

Delmas and colleagues demonstrates that, lamellar structure AMO_2 materials can be categorized into two primary categories: P2 and O3 layered structures [7]. Specifically, the letters P and O denote the occupied sites by alkali metal ions in the crystallographic structure (where P corresponds to the trigonal prismatic coordination, and O indicates octahedral coordination). Number 2, 3 indicates the number of MO_2 slabs per unit cell. The P2 and O3-type layered structures are more favorable for sodium insertion and deintercalation during electrochemical cycling in comparison with the P3 and O2 structures [8].

Among AMO_2 type layered oxides, sodium-manganese-based materials have emerged as promising candidates for sodium-ion battery cathodes. Some layered transition metal oxides have been reported, for example, $Na_{0.6}MnO_2$ delivers an initial capacity of approximately 155 $mAh.g^{-1}$, but suffers from a capacity loss of approximately 55% after only 10 cycles [9]. The $Na_{0.44}MnO_2$ oxide exhibits a specific capacity of 140 $mAh.g^{-1}$ when used as a cathode material in sodium ion battery [10]. The $NaNiO_2$ oxide achieves an initial capacity of 131 $mAh.g^{-1}$ at a current density of 10 $mA.g^{-1}$, with a capacity loss of approximately 15% observed after the first 5 cycles [1]. Nanostructured $Na_{0.7}MnO_2$ delivers an excellent capacity of up to 135 $mAh.g^{-1}$ at a current density of 10 $mA.g^{-1}$ but retains less than 50% of its initial capacity after 100 cycles [11].

The aforementioned materials reveal that the poor cycling stability is the main obstacle of Na_xMO_2 type materials. The larger size of sodium ion and the substantial volume change in the layered oxide structure during sodium ions insertion and extraction are the primary reasons of this challenge [8] - [12]. A large number of studies are executed to increase the cycling performance of positive electrode materials, mainly focusing on the addition various transition metals. For examples, (i) $Na_xFe_{1/2}Mn_{1/2}O_2$ oxide provides a specific capacity of 190 $mAh.g^{-1}$ and retains of 150 $mAh.g^{-1}$ after 30 cycles [13]; (ii) $Na_{2/3}Ni_{1/3}Mn_{2/3}O_2$ oxide achieves a specific capacity of 150 $mAh.g^{-1}$ and retaining 70% of its capacity after 30 cycles; (iii) The Co and Cu doped $Na_{2/3}Mn_{1/2}Co_{1/3}Cu_{1/6}O_2$ material has a capacity of 118 $mAh.g^{-1}$ at charge-discharge current density 10 $mA.g^{-1}$, and the capacity maintains 66% after 100 cycles [14]. Additionally, Liangtao Yang and colleagues successfully synthesized P2-$Na_{0.67}Mn_{0.6}Ni_{0.2}Li_{0.2}O_2$, in which a part of Mn ions were partially replaced with Li^+ anh Ni^{2+} ions. The P2- $Na_{0.67}Mn_{0.6}Ni_{0.2}Li_{0.2}O_2$ delivers a capacity of 115 $mAh.g^{-1}$ and 80 $mAh.g^{-1}$ under current rates of 10 $mA.g^{-1}$ and 100 $mA.g^{-1}$, and the capacity exhibited a retention of 95.6% upon 100 cycles under 10 $mA.g^{-1}$ rate [15].

Our previous research validated the electrochemical properties of P2-$Na_{2/3}Li_{1/3}Mn_{0.95}Co_{0.05}O_2$ oxide. The synthesized sample has a specific capacity of 112 mAh.g^{-1} charged-dischrged at a current density of 10 mA.g^{-1} and superior capacity retention of 97.7% over 100 cycles at the same current rate [16]. In another case, the P2-$Na_{0.8}Li_{0.1}Mn_{0.8}Co_{0.1}F_{0.1}O_t$ was synthesized by the sol-gel method, which exhibited a electrochemical capacity of 135.51 mAh.g^{-1} under a current density of 8 mA.g^{-1} and its capacity kept above 100 mAh.g^{-1} despite charge-discharge at fast current rate of 100 mA.g^{-1}. This compound retained 92.69% of the initial value upon 100 charge-discharge cycles [17]. Additionally, the P2-$NaZn_{0.2}Mn_{0.8}O_2$ material had a superior initial discharge capacity of 155 mAh.g^{-1} within the voltage range of 1.5-4 V under a current rate of 10 mA.g^{-1}. the specific capacity kept at 100 mAh.g^{-1} over 50 cycles [18].

According to results of reported studies, replacing a part of Mn content by using alternative transition metals can improve bettery performance of Na-Mn-based oxide. In this research, our group successfully fabricated Ni-doped Na-Li-Mn compound (NLMN) with a layered crystal structure to be used as cathode material for Na-ion battery. The addition of Li^+ anh Ni^{2+} ions into the P2-type Na-Mn-O_2 structure provides specific structural and electrochemical benefits. Lithium ions does not involve in electrochemical reactions but they are prone to occupy transition metal positions and play the role in stabilization by reducing Jahn-Teller distortions, which caused by Mn^{3+}, thus improving structural stability during cycling. On the other hand Nickel ions are electrochemically active species, they play the role in electrochemical reactions and enhance electronic conductivity. Furthermore, Ni doping in the compound may increases voltage window and decreases interfacial change transfer resistance. The synergistic effect of Li and Ni substitution leads to enhance capacity stability and rate performance in NLMN material.

The analyses of the morphological, structural, and electrochemical properties of the NLMN materials show that the synthesized NLMN achieves a capacity of 114.11 mAh.g^{-1}. This ressult is higher than that of $Na_{0.74}Co_{0.95}Nb_{0.05}O_2$ material (91 mAh.g^{-1}) [19] and of $Na_2MnP_2O_7$ material (80 mAh.g^{-1}) [20]. The cycling performance of synthesized NLMN surpasses that of $Na_{0.6}MnO_2$ material [9], $NaNiO_2$ material [1] and $Na_{0.7}MnO_2$ material [11]. Furthermore, the dual Li and Ni doping strategy provides a promising platform for further optimization. These results suggest that the synthesized NLMN material can be developed for use as a cathode in sodium ion battery.

2. Experiments

2.1. Synthesis of NLMN materials

Nickel-doped Na-Li-Mn oxide (NLMN) was prepared via solid state reaction. The raw materials: Na_2CO_3 (≥ 99.5%, Sigma-Aldrich), Li_2CO_3 (≥ 99.0%, Sigma-Aldrich), $Ni(OH)_2$ (≥ 99.9%, Sigma-Aldrich), and $MnCO_3$ (≥ 99.9%, Sigma-Aldrich), were mixed in a molar ration of Na:Li:Mn:Ni = 1:0.15:0.8:0.2. The mixture was first manually ground using motar and pestle and followed by mixing via ball-milling at a rotation speed of 120 rpm over 5 h. The prepared uniform mixture was calcined under appropriate thermal conditions via two stages. (i) the precursor mixture was initially heat treated at 550 °C for 6 h in air to decompose carbonate; (ii) The compound was subsequently calcined at a high temperature of 850 °C for 24 h to produce the $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$ material (NLMN). The reaction leading to NLMN upon calcination is described by the equation below:

$$5Na_2CO_3 + 8MnCO_3 + \frac{3}{4}Li_2CO_3 + 2Ni\left(OH\right)_2 + \frac{9}{4}O_2 = 10Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2 + \frac{55}{4}CO_2 + 2H_2 \quad (1)$$

2.2. Material characterizations

The crystal structure of NLMN was characterized via X ray diffraction (XRD, using a Siemens D5005 diffractometer with Cu-Kα source) using radiation (λ = 1.54 Å) over the 2θ range of 10° - 70°.

The surface morphology of NLMN material was examined using electron microscopy equipped with an EDS detector (SEM-EDS, JEOL JSM-6490).

The electrochemical characteristics of synthesized NLMN was tested via charge–discharge measurement under constant current (GCD, NEWARE battery tester). The charge/discharge capacity at different current densities in the voltage window of 1.5 - 4 V was evaluated via a NEWARE battery tester (BTS). Electrochemical measurements were performed on a CR2032-type sodium-ion battery, where the cathode was the NLMN and the anode was sodium foil. The CR2032 coin cells were assembled in a glove box with oxygen and moisture levels less than 0.1 ppm and stabilized for 24 h before electrochemical characteristics testing.

To produce cathode, the slurry was prepared by mixing NLMN, black carbon, and polyvinylidene fluoride used as the binder with a weight proportion of 80:10:10 using N-methyl-2-pyrrolidone as the solvent. Afterwards, the slurry was covered evenly on aluminum foil with a thickness of 15 µm and dried under vacuum condition at 100 °C for 24 h and then punched into round electrodes for use in CR2032 cells.

3. Results and discussions

3.1. Structure characteristics

Figure 1 describes XRD pattern of the synthesized NLMN compound. Based on the standard pattern (JCPDS 27-0751), the major diffraction peaks correspond to a hexagonal P2 -type phase, belonging to space group P63/mmc. The symbol "p" refers to the prismatic positions occupied by sodium ions, and the digit "2" indicates two MnO_2 layers within the unit cell. In the P2-type structure, Na^+ ions reside between MnO_2 layers composed of edge sharing MnO_6 octahedra and residing in two crystallographically distinct prismatic sites [21].

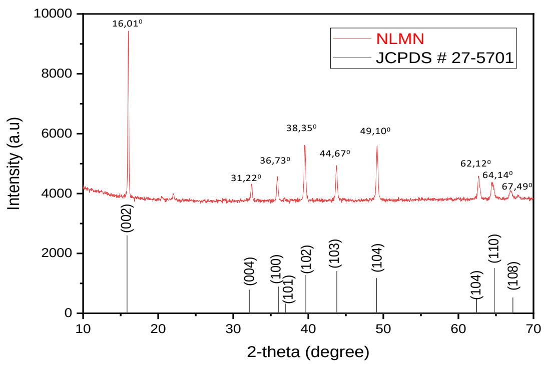

Figure 1. *XRD profile of NLMN material*

The XRD reflections detected at 16.01°, 31.22°, 36.73°, 38.35°, 44.67°, 49.10°, 62.12°, 64.14° and 67.49° correspond to (002), (004), (100), (102), (103), (104), (106), (110) and (008) planes, respectively. The diffraction results confirm single-phase formation of the synthesized product. The observed XRD peaks are consistent with the data reported in Li-doped $NaLi_{0.2}Mn_{0.8}O_2$ material, Ni-doped $NaLi_{0.2}Ni_{0.25}Mn_{0.75}O_2$ oxide, Co-doped $Na_{2/3}Li_{1/3}Mn_{0.95}Co_{0.05}O_2$ oxide [16]. Ti-doped $Na_{1.0}Li_{0.2}Mn_{0.7}Ti_{0.1}O_2$ oxide [21], Zinc-doped sodium manganese oxide $NaZn_{0.2}Mn_{0.8}O_2$ material [18].

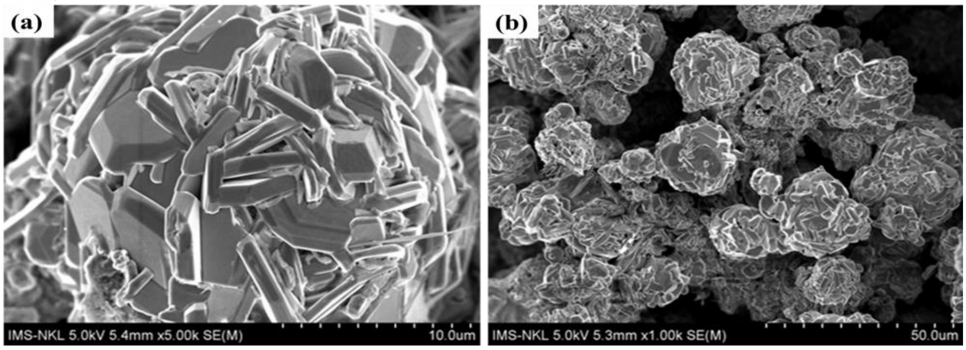

Figure 2. *SEM images of NLMN material*

The surface morphology of the NLMN sample is analyzed by scanning electron microscopy (SEM) and the result is shown in Figure 2. Figure 2a and 2b are surface images of the same area of the synthesized NLMN compound at different magnifications. Figure 2a captured at a magnification of 5000, revealed that the material has polyhedral - shaped particles. Figure 2b with magnification of 1000 illustrates a particle agglomeration observed on a large area. The results show that, the synthesized NLMN has a polyhedral form with particle sizes in the range of 2 - 5 μm and loosely aggregated. The variation in particle size and particle agglomerates in prepared NLMN compound by the solid state method has also been reported in earlier studies [15], [22].

The constituent elements in the synthesized NLMN compound is analyzed by EDS technique. Atomic percentage values are employed to determine the stoichiometric ratio of the elements. This element component is depicted in Figure 3. The detected elements in NLMN material include Na, Mn, Ni, and O, which correspond to the raw materials.

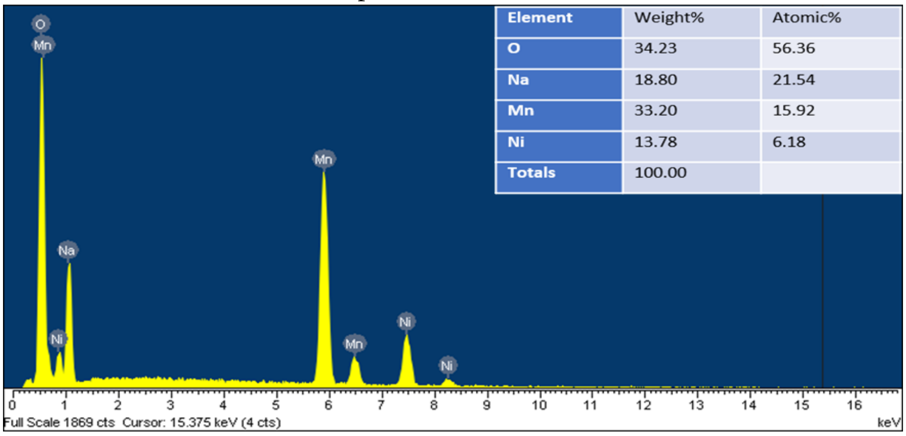

Figure 3. *EDS spectrum of NLMN material*

The lithium element is absent in the EDS results because the EDS technique is not capable of detecting elements with low atomic masses, such as lithium. The elemental ratio of Na, Mn, Ni, and O is found to be 1:0.8:0.2:2, which corresponds to the precursor salts.

3.2. Electrochemical performance

Figure 4 shows the GCD plots of the NLMN compound measured at a current density of 10 mA.g-1 rate in the potential window of 1.5 - 4.5 V. The initial three charge-discharge cycles coincide closely which suggests high cycling efficiency of the synthesized NLMN sample. As shown in Fig. 4, the charge/discharge capacities during the first three cycles of NLMN compound were recorded as 127.29/114.11, 134.08/113.75, and 120.88/113.64 mAh.g^{-1}, respectively. The result confirms that the charging and discharging processes of NLMN material are highly reversible in the first cycle and gradually enhanced in the coming cycles. The discharge specific capacity of NLMN material is superior to that of $Na_{0.74}Co_{0.95}Nb_{0.05}O_2$ material (91 mAh.g^{-1}) [19] and $Na_2MnP_2O_7$ material (80 mAh.g^{-1}) [20]. The voltage profile exhibits a gradual slope between 2.5 and 4 V, implying that the capacity was mainly contributed in the voltage range of 2.5 - 4 V.

The cycling ability of the prepared material was further examined via repeated charge-discharge with constant current. Figure 5 depicts the capacities over 50 cycles of the NLMN compound at a current density of 10 mA.g^{-1} rate. As shown in Figure 5, the first discharge specific capacity of NLMN material at a current density of 10 mA.g^{-1} was 114.11 mAh.g^{-1}. The specific discharge capacity of NLMN upon 50 cycles was 65.44 mAh.g^{-1}. The capacity retains approximately 57.2% of the first cycle discharge capacity. The loss of capacity after 50 cycles could be mainly caused by structural degradation as Na^+ ions are inserted into and released from the cathode material, the dissolution of transition metal into electrolyte and formation of non-reversible by-products. These deterioration processes are typical in P2-structured layered cathodes. To address these issues, It is necessary to continue doing research to improve the material characteristics by changing the element content or doping other multivalent transition metals (e.g., Mg^{2+}, Cu^{2+}, Ti^{4+}) to improve structural robustness and minimize Jahn-Teller deformation or applying surface coatings (Al_2O_3, carbon layer) to improve interfacial stability, which can improve the cycling performance of the material.

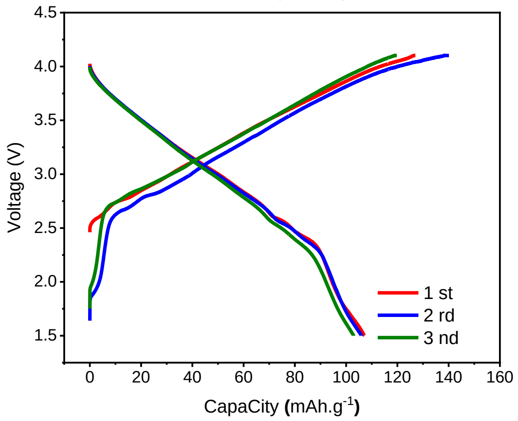

Figure 4. *The charging/discharging curves of NLMN at a current density of 10 mA.g^{-1}*

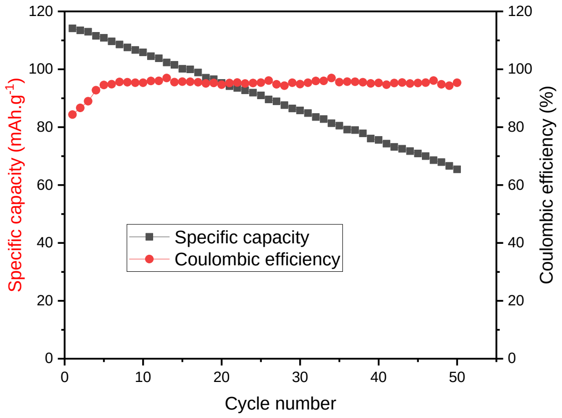

Figure 5. *The charging-discharging profiles of NLMN at 10 mA.g^{-1} for 50 cycles*

Although the cycling performance of synthesized NLMN material is limited, it still surpasses certain earlier reported P2-type cathode compounds [1], [9], [11]. Additionally, Figure 5 reveals that the Coulombic efficiency of NLMN compound remains consistent over 20 cycles. The Coulombic efficiency of NLMN ranges between 95% and 97%. This value is higher than several literature reported about P2-phase layered oxide cathode [15], [22] - [24].

The rate capability of NLMN materials was evaluated by cycling at various current densities of 10 mA.g^{-1}, 20 mA.g^{-1}, 30 mA.g^{-1}, 40 mA.g^{-1}, 50 mA.g^{-1}, and the results are presented in Figure 6. The results show that the discharge capacity of NLMN material decreases as the current rate increases. Specifically, the capacity decreases significantly from 114.11 mAh.g^{-1} at 10 mA.g^{-1} to 6.27 mAh.g^{-1} at 40 mA.g^{-1}. When the cell is cycled at 50 mA.g^{-1}, the discharge capacity of NLMN falls below 5 mAh.g^{-1}. However, when the current density is backed to 10 mA.g^{-1}, the capacity recovers to a level of 102 mAh.g^{-1}.

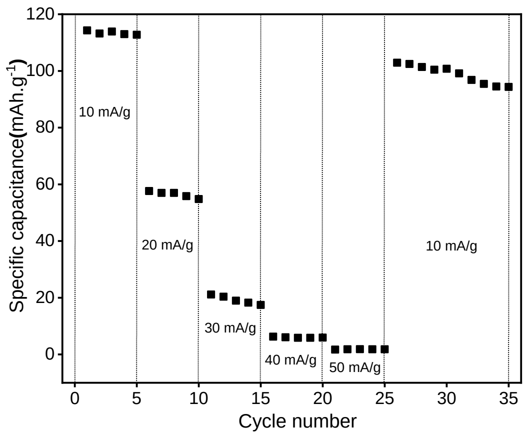

Figure 6. *The charge-discharge results of NLMN at multiple current rate*

This recovery indicates that the material maintains good structural integrity after high – rate cycling. The material exhibits high capacity when cycled at a low rate and exhibits poor capacity at elevated current densities, this phenomenon can be attributed to limited intercalation time for the Na^+ embedding into the crystal structure; therefore, electrochemical activity is limited on the surface of the electrode material [22], [25].

Although electrochemical impedance spectroscopy (EIS) tecnique was not performed in this research due to instrumental limitations, the electrochemical performance was studied through galvanostatic charge-discharge curves and rate performance measurements. The decline in capacity and limited rate capacity could be elevated interfacial resistance and slow Na^+ diffusion kinetics during repeated cycling, which are typical of P2-type cathodes. Further studies will focus on electrochemical impedance spectroscopy (EIS) analysis and CV measurements to gain a deeper understanding about these mechanisms.

4. Conclusions

Ni substituted Na-Li-Mn oxide (NLMN) was successfully obtained via a typical solid state reaction. XRD results indicate that the synthesized NLMN sample corresponds to the hexagonal layered P2-type structure of $Na_{0.7}MnO_2$ (JCPDS-270751) phase. SEM analysis reveals that individual particle size falls within 2 - 5 μm and loosely connected. The EDS spectrum indicated that the combinatorial formula of NLMN material was found be $Na_{1.0}Li_{0.15}Mn_{0.8}Ni_{0.2}O_2$. The electrochemical characteristics of NLMN compound were thoroughly investigated via galvanostatic cycling at both fixed and variable current densities. The synthesized NLMN sample delivers a large of charge specific capacity of 127.29 $mAh.g^{-1}$ in the first cycle at a current density of 10 $mA.g^{-1}$. Coulombic efficiency is about 95-97%. However, the cycle performance of NLMN material is still limited. After 50 cycles, the remaining capacity is only about 57.2% of the discharge capacity of the first cycle at 10 $mA.g^{-1}$ rate. Therefore, it is necessary to study further to improve the cycling performance of the NLMN material before applying this material in sodium ion battery as a cathode.

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