

SYNTHESIS AND ELECTROCHEMICAL CHARACTERISTICS OF ZINC-DOPED SODIUM MANGANESE OXIDE AS A CATHODE MATERIAL FOR SODIUM-ION BATTERIES

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Abstract

In this article, zinc-doped sodium manganese oxide (NZMC) was synthesized by a conventional solid-state reaction method. Structure and morphology of the NZMC material were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS). The results show that Zn was successfully doped into the sodium manganese-based oxide (NMC) material. The NZMC material was utilized as a cathode material in CR2032-type coin cells for testing its electrochemical characteristics. The NZMC material had a superior initial discharge capacity of 155 mAh.g⁻¹ between 1.5-4 V at a current density of 0.1 C. The capacity remained at 100 mAh.g⁻¹ after 50 cycles. The results suggest that the NZMC material is a promising cathode for sodium-ion batteries.

Keywords: *Sodium-ion battery; sodium manganese oxide; zinc-doped sodium manganese oxide; solid-state reaction.*

1. Introduction

Lithium-ion batteries are commonly used in electronic devices because of their advantages such as: light weight, low redox potential ($E_{\text{Li}^+/\text{Li}} = -3.05 \text{ V}$) compared to standard hydrogen electrodes, small radius of Li^+ ions, long cycle life, and high energy efficiency. The utilization of batteries for electric vehicles and other mobile equipment is increasing, which leads to a high demand for lithium metal. Even though many manufacturers have tried to recycle lithium from damaged batteries, the lithium supply does not match the demand in battery usage [1-3]. Moreover, the cost of lithium metal is rapidly increasing due to the scarcity of lithium sources. Sodium-ion batteries are a promising candidate to replace lithium-ion batteries with outstanding advantages such as: (i) low cost of sodium, (ii) low redox potential ($E_{\text{Na}^+/\text{Na}} = -2.71 \text{ V}$) compared to the standard hydrogen electrode, (iii) low toxicity, and (iv) easy synthesis [4, 5].

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The structure and operating principle of sodium-ion and lithium-ion batteries are similar. However, the radius of sodium ion is larger than lithium ion, so the ability of sodium ion to intercalate into and extract from the structure of electrode materials is less than that of lithium ion. Therefore, finding electrode materials with structure suitable for sodium ion intercalating and extracting is essential. There are reported cathode materials meeting the requirement such as: AMO_2 , AMPO_4 (i.e., A is alkali metals and M is transition metals), and phosphates polyanions [6, 7].

Among AMO_2 -type materials, sodium-manganese-based materials have a great potential for application in sodium-ion batteries. There are a number of sodium-manganese-based materials with different structures suitable for sodium ion intercalation and extraction. For example, the tunnel-structured $\text{Na}_{0.44}\text{MnO}_2$ ($\text{Na}_4\text{Mn}_9\text{O}_{18}$) material has been reported as a cathode material with a charge-discharge capacity of 140 mAh.g^{-1} [2-6]. AMO_2 -type oxides with a layered structure have been widely investigated. AMO_2 oxides can be divided into two main groups, which are called as P_n and O_n structures according to Delmas' notation [4-8]. Specifically, P_n , O_n ($n = 2,3$) with P (triangular prism) and O (octahedron) indicate the position occupied by sodium ions in the structure. The P_2 and O_3 structure are more suitable for sodium ions insertion and extraction during the charge-discharge process compared to the P_3 and O_2 structure.

Recent studies have reported that NaMnO_2 materials can insert and extract 80% of its sodium ions amount during the charge/discharge processes. They achieve a capacity of 185 mAh.g^{-1} at the first cycle with a charge-discharge current density of 15 mA.g^{-1} but the capacity decreases rapidly. The capacity retains a value of 132 mAh.g^{-1} after 20 cycles [4, 5, 9]. Transition metal doping can improve the cyclability of NaMnO_2 materials. For example: (i) $\text{Na}_x[\text{Fe}_{1/2}\text{Mn}_{1/2}]\text{O}_2$ material achieves a capacity of 190 mAh.g^{-1} at charge-discharge current density of 12 mA.g^{-1} between 1.5-4.3 V at the first cycle and the capacity retains at 150 mAh.g^{-1} after 30 cycles [8, 10]; (ii) $\text{P}_2\text{-Na}_{2/3}\text{Ni}_{1/3}\text{Mn}_{2/3}\text{O}_2$ material has a capacity of 150 mAh.g^{-1} at charge-discharge current density of 10 mA.g^{-1} between 2-4.5 V and the capacity retains 70% after 30 cycles; (iii) Pang et al. reported that Co and Cu doped $\text{P}_2\text{-Na}_{2/3}\text{Mn}_{1/2}\text{Co}_{1/3}\text{Cu}_{1/6}\text{O}_2$ 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 [11]. Our previous works showed the electrochemical performance of $\text{P}_2\text{-Na}_{1.0}\text{Li}_{0.2}\text{Mn}_{0.7}\text{Ti}_{0.1}\text{O}_2$ material, which was synthesized by solid-reaction method [12, 13]. The material achieves a capacity of 163 mAh.g^{-1} at a charge-discharge current density of 15 mA.g^{-1} (0.1 C) between 1.5-4 V. The capacity maintains 90% after 50 cycles. When charging and discharging at a high current density of 150 mA.g^{-1} (1 C), the material achieves the capacity of 100 mAh.g^{-1} .

Based on the results of published works, replacement of partial manganese by other transition metals can enhance the electrochemical performance of sodium manganese based oxide. In this work, we chose to synthesize zinc-doped sodium manganese oxide and investigated the characteristics of the material as a cathode for sodium-ion battery. The results showed that the synthesized material achieved a superior rate capability and cyclability.

2. Experiment

A mixture of sodium carbonate (Na_2CO_3 , Sigma-Aldrich), zinc oxide (ZnO , Sigma-Aldrich), manganese carbonate (MnCO_3 , Sigma-Aldrich) was fixed at a molar ratio such that $\text{Na}:\text{Zn}:\text{Mn} = 1:0.2:0.8$. The mixture was carefully ground using mortar and pestle for 30 mins and then ball-milled at 100 rpm for 2 h. The obtained homogeneous mixture was calcined at appropriate temperature through 2 steps: the mixture was first heat-treated at 550°C for 8 h in the air to remove carbonate in transition metal salts and then they were calcined at 800°C for 10 h to obtain the $\text{NaZn}_{0.2}\text{Mn}_{0.8}\text{O}_2$ material (NZMC). The reaction expected for $\text{NaZn}_{0.2}\text{Mn}_{0.8}\text{O}_2$ was:



The structure and morphology characteristics of NZMC were confirmed by X-ray diffraction (XRD, Siemens D5005 X-ray diffractometer with $\text{Cu-K}\alpha$ radiation), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS, JEOL JSM-6490).

The electrochemical performance of NZMC was evaluated through cyclic voltammetry (CV, Ivium V55647) and electrochemical impedance spectroscopy (EIS, Ivium V55647). The charge/discharge capacity at different current densities between 1.5-4.5 V was measured on a NEWARE battery testing system (BTS). Electrochemical measurements were performed on CR2032-type coin cells, in which the cathode was NZMC and the anode was sodium foil, polypropylene (PE) film as the separator and a solution of 1M NaPF_6 in ethylene carbonate/diethylene carbonate (EC/DEC, 1:1 by volume) as the electrolyte. The CR2032 coin cells were assembled in glove box with oxygen and humidity less than 0.1 ppm and maintained stable for 24 hours before electrochemical characteristics testing.

To prepare the cathode, a slurry was formed by mixing active material NZMC, carbon black (super P) and polyvinylidene fluoride (PVDF) as a binder by weight ratio of 8:1:1 in N-methyl-2-pyrrolidone (NMP). The slurry was evenly coated on aluminum foil. The coated tape was dried at 100°C for 24 hours in a vacuum and then cut into standard diameter circles for assembling CR2032 cells.

3. Results and discussions

3.1. Structure characteristics

Figure 1 shows the X-ray diffraction of synthesized material ($\text{NaZn}_{0.2}\text{Mn}_{0.8}\text{O}_2$). The XRD pattern of $\text{NaZn}_{0.2}\text{Mn}_{0.8}\text{O}_2$ (NZMC) material can be attributed to the hexagonal P_2 -structure, space group of $P63/mmc$ of $\alpha\text{-Na}_{0.70}\text{MnO}_{2.05}$ (JCPDS No 27-0752), which suggests that zinc ions partially replaced manganese ions in the lattice structure. The P letter indicates the prismatic sites occupied by Na^+ ions and the number “2” stands for the two MnO_2 sheets contained in the unit cell. In this structure, sodium ions are sandwiched between MnO_2 slabs made of edge-sharing MnO_6 octahedral sites and located at two distinct trigonal prismatic sites [14]. The main diffraction peaks observed at 16.01° , 31.22° , 36.73° , 38.35° , 44.67° , 49.10° , and 65.02° are properly identified to the (002), (004), (100), (101), (102), (103), (104), and (110) planes, respectively. The other peaks marked by an asterisk are attributed to the secondary phase of $(\text{Mn}_{0.848}\text{Zn}_{0.147})\text{Mn}_{1.999}\text{O}_2$ (JCPDS No 01-007-9105). The main diffraction peaks are similar to the results published for lithium-substituted hexagonal P_2 -structure $\text{NaLi}_{0.2}\text{Mn}_{0.8}\text{O}_2$, nickel substituted manganese $\text{NaLi}_{0.2}\text{Ni}_{0.25}\text{Mn}_{0.75}\text{O}_2$ material [15], cobalt doped sodium manganese oxide [16], titanium doped sodium manganese oxide [17].

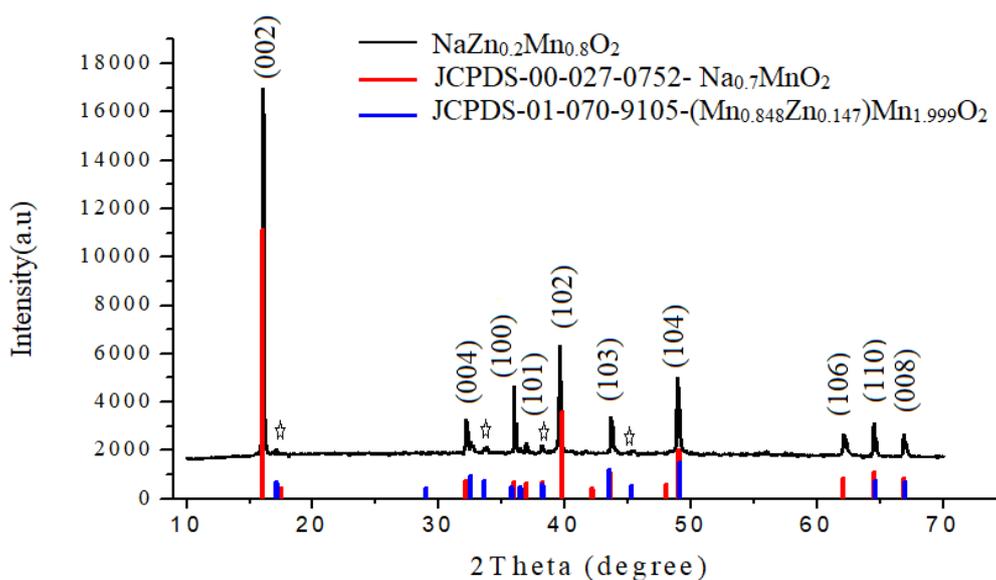


Fig. 1. XRD pattern of NZMC material.

The morphology of NZMC material is confirmed by scanning electron microscopy (SEM) (Fig. 2). The NZMC particles have polyhedron shape with various

sizes ranging from 500 nm to 2 μm . The large particle size of materials synthesized by solid-state reaction method is also reported in previous studies [10, 14, 15].

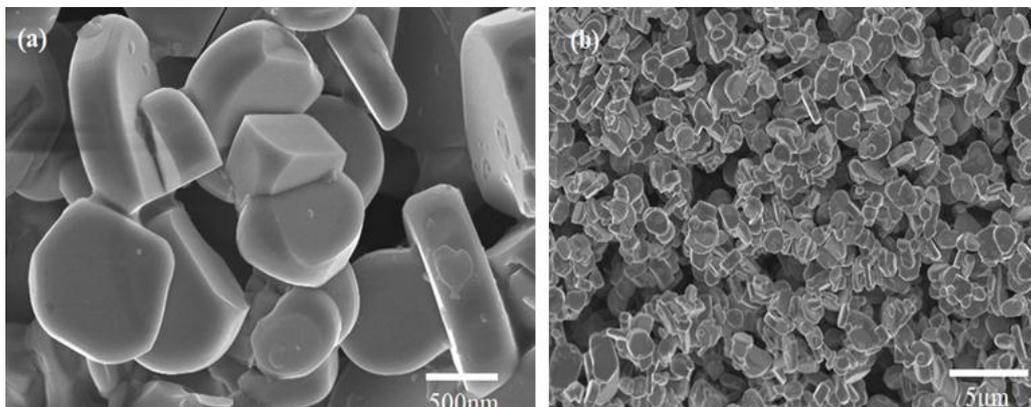


Fig. 2. SEM images of NZMC material.

The composition of the NZMC material was evaluated by energy dispersive X-ray spectroscopy (EDS) method (Fig. 3). The result shows that the synthesized NZMC material contained Na, Zn, Mn, and O elements. The atomic ratio between the Na, Zn, Mn, and O elements is approximately 1:0.2:0.8:2, which is consistent with the initially used raw precursor ratio.

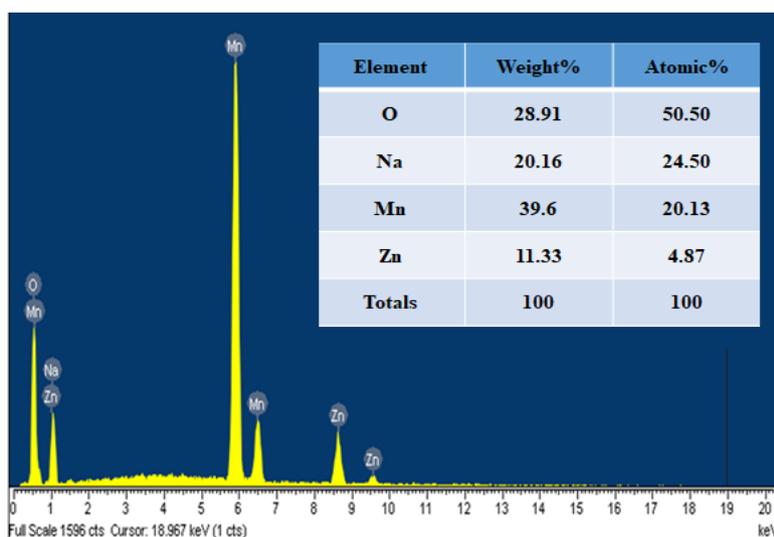


Fig. 3. Energy dispersive X-ray spectroscopy (EDS) result of NZMC.

3.2. Electrochemical characteristics

Figure 4a is charge/discharge profiles of the NZMC material at the current density of 0.1 C with voltage range from 1.5 to 4 V. The first charge/discharging curves are overlapped, which suggests an outstanding cyclability of the NZCM material. The first charge capacity and discharge capacity are approximately 155 $\text{mAh}\cdot\text{g}^{-1}$ and 150 $\text{mAh}\cdot\text{g}^{-1}$,

respectively. The charge-discharge voltage slowly changes in the voltage range of 1.75-2.5 V, indicating that the largest contribution of capacity in the charge-discharge process occurred between 1.75-2.5 V.

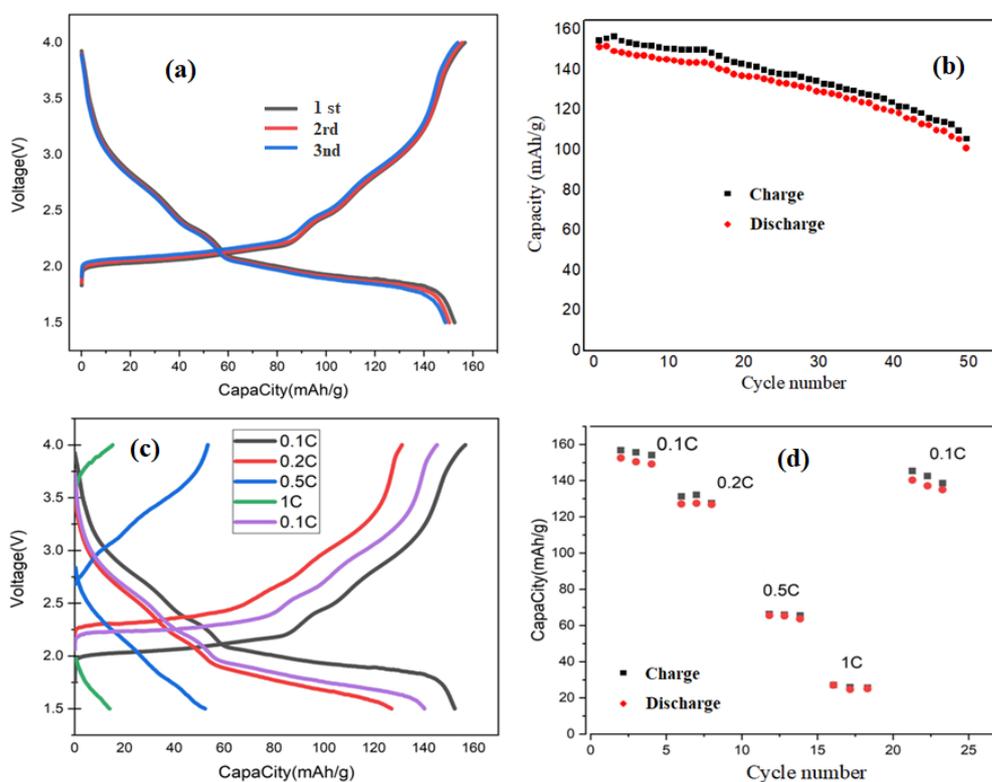


Fig. 4. (a, b) - The charging-discharging profiles of NZMC at 0.1 C;
(c, d) - The charging-discharging profiles at different current densities.

Figure 4b shows the charge-discharge capacity for 50 cycles of the NZMC material at a current density of 0.1 C. The specific capacity of the material gradually decreases to approximately 100 mAh.g^{-1} at the 50th cycle. The capacity remains about 65% of its first cycle. The cyclic efficiency of the synthesized material is higher than that of reported manganese dioxide-based cathode materials [1, 2, 18].

Figures 4c and 4d illustrate the charge-discharge curves and specific capacity of NZMC material at current densities of 0.1 C, 0.2 C, 0.5 C, and 1 C. The capacity decreases significantly from 155 mAh.g^{-1} at 0.1 C to 20 mAh.g^{-1} at 1 C. When the current density is backed to 0.1 C the capacity regains the value of 140 mAh.g^{-1} , which means the structure of NZMC material is stable after cycling at high current densities. The material has high capacity when charging/discharging at low current density and low capacity when charging/discharging at high current density. In other words, the

internal resistance of the material is increased when increasing the charge-discharge current density. When charging and discharging at high current densities, the voltage increases rapidly during charging and decreases quickly during discharging (Fig. 4c).

The cyclic voltammetry (CV) scanning is shown in Fig. 5a. The CV scanning was carried out in the range from 1.5 to 4 V with a scanning speed of $0.1 \text{ mV}\cdot\text{s}^{-1}$ on an Ivium V55647 machine, with the working electrode connected to the positive terminal of the battery and counter and reference electrodes connected to the negative terminal of the battery.

On the CV spectrum, there are 3 pairs of redox peaks, which correspond to the three voltage plateaus in the charge-discharge processes. The first pair of redox peaks appeared at about 1.75 V and 2.5 V, which correspond to the oxidation of Zn to Zn^{2+} and the reduction of Zn^{2+} to Zn, respectively. The current density of the first pair of redox peaks is the highest density among of the 3 pairs. The first pair of redox peaks is corresponded to the longest potential plateaus in the charge-discharge curves (Fig. 4a), where the main capacity is delivered. The second pair of redox peaks appeared at 2.25 V and 3.25 V, which correspond to the oxidation of Mn^{2+} to Mn^{3+} and the reduction of Mn^{3+} to Mn^{2+} , respectively. The third pair of redox peaks appeared at 2.75 V and 4 V, which corresponded to the oxidation of Mn^{3+} to Mn^{4+} and the reduction of Mn^{4+} to Mn^{3+} .

The current densities of the second and the third redox peak pairs are smaller than that of the first one (Fig. 5a). This proves that doping Zn into the sodium manganese-based dioxide material system increased the ability of Na^+ ions to intercalate/extract into/from the structure of the material. This also proves that the main capacity of the NZMC material was contributed to the Zn/Zn^{2+} redox reaction.

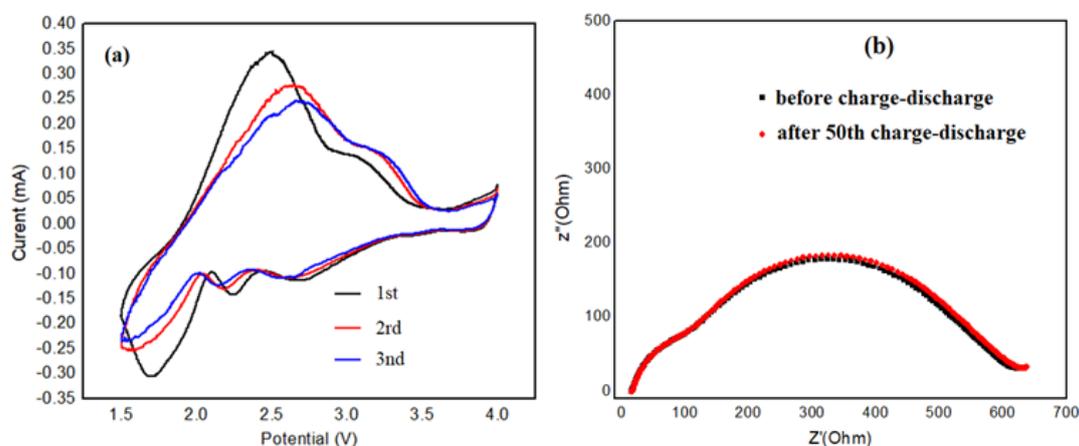


Fig. 5. (a) The CV curves of NZMC at $0.1 \text{ mV}\cdot\text{s}^{-1}$; (b) Nyquist plots of the NZMC materials.

The EIS characterization was conducted in a frequency range from 1 MHz to 0.1 Hz to evaluate the electrochemical reaction kinetics. The Nyquist plots of NZMC material before charge-discharge and after 50 charge-discharge cycles are shown in Fig. 5b. The EIS spectra before and after 50 cycles charge-discharge have a slight difference. The total impedance is also relatively low, which proves that Na⁺ ions are reversibly intercalated and extracted into/from the NZMC material structure during cycling.

4. Conclusions

The layered structure NZMC material was successfully synthesized by a conventional solid-state reaction method. The as-prepared material is related to the hexagonal P₂-structure of Na_{0.7}MnO₂ (JCPDS - 270752) phase. The EDS result confirmed that NZMC material has the approximate combinatorial formula: NaZn_{0.2}Mn_{0.8}O₂. The electrochemical properties of NZMC material were carefully and systematically evaluated by means of CV, charge-discharge with constant current density and EIS. The NZMC material has a high specific capacity of 155 mAh.g⁻¹ at first. The capacity remains about 100 mAh.g⁻¹ after 50 cycles. The results suggest that the NZMC material is a potential positive electrode material for sodium ion batteries.

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TỔNG HỢP VÀ NGHIÊN CỨU ĐẶC TÍNH ĐIỆN HÓA CỦA VẬT LIỆU CATỐT NATRI MANGAN ĐIOXIT PHA TẠP KẼM ỨNG DỤNG CHO PIN NATRI-ION

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Tóm tắt: Bài báo trình bày kết quả quá trình tổng hợp vật liệu natri mangan đioxit pha tạp kẽm (NZMC) bằng phương pháp phản ứng pha rắn qua hai bước xử lý nhiệt ở 550°C và 800°C. Vật liệu NZMC sau tổng hợp được nghiên cứu đặc trưng cấu trúc bằng phương pháp nhiễu xạ tia X (XRD) và SEM-EDX. Kết quả cho thấy đã pha tạp thành công Zn vào vật liệu trên nền natri-mangan đioxit (NMC). Đặc tính điện hóa của vật liệu (NZMC) đã tổng hợp được nghiên cứu bằng phương pháp tạo lớp phủ lên lá nhôm sau đó cắt, ghép thành pin tiêu chuẩn CR232 với cực âm là natri kim loại (các thao tác được tiến hành với môi trường khí argon trong glovebox). Pin hoàn thiện được phóng nạp với các tốc độ nạp, xả khác nhau trên hệ thống nạp-xả đa kênh (Battery Testing System (BTS)) cho kết quả đạt được 155 mAh/g ở chu kỳ đầu và 100 mAh/g ở chu kỳ 50 với tốc độ nạp-xả 0,1 C trong khoảng thế từ 1,5 V đến 4 V. Tổng trở điện hóa của pin (EIS) và quét thế vòng tuần hoàn (CV) được đo trên máy đo điện hóa đa năng (Ivium V55647) để kiểm tra các tính chất điện hóa của vật liệu (NZMC) đã tổng hợp được. Kết quả CV cho thấy sự xuất hiện 3 cặp pic ứng với các quá trình chuyển điện tích trong pin. Vật liệu tổng hợp được có triển vọng khả quan để ứng dụng trong pin natri-ion.

Từ khóa: Pin natri-ion; natri mangan đioxit; natri mangan đioxit pha tạp kẽm; phản ứng pha rắn.

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