

A HIGH-ENERGY ANODE FOR SODIUM-ION BATTERIES BASED ON SODIUM MOLYBDATE MATERIALS

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Abstract

In this study, sodium molybdate (Na_2MoO_4) was synthesized by a conventional solid-state reaction method. Crystal structure and morphology of the Na_2MoO_4 material were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS). The Na_2MoO_4 material was utilized to fabricate CR2032-type coin cells, and later evaluated for its electrochemical characteristic. The electrochemical characteristics of Na_2MoO_4 were evaluated through cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and charging-discharging at different current densities on a NEWARE battery testing system (BTS). The results of studying the electrochemical properties of Na_2MoO_4 material has shown that it has pretty good electrochemical properties. In particular, the Na_2MoO_4 electrode material has achieved an outstanding specific capacity of about 420 mAh.g^{-1} at 0.1 C within the voltage range from 0.2 to 2.2 V. The Na_2MoO_4 material exhibited a quite good cycling performance, it is able to remains up to 80% specific capacity over 50 cycles at the current density of 0.1 C. In addition, when charge/discharge at the high current density of 1 C, it has retained a specific capacity of about 240 mAh.g^{-1} . The results suggest that the Na_2MoO_4 material is a promising anode for sodium-ion batteries.

Keywords: Sodium-ion battery; sodium molybdate; anode materials; solid-state reaction.

1. Introduction

The increasing power demand for flexible electronics stimulates the development of flexible batteries with high performance, light weight, and low cost. However, the popularity of lithium-ion batteries has meant that lithium are demanded increasingly, and 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 low cost of sodium, low redox potential ($E_{\text{Na}^+/\text{Na}} = -2.71 \text{ V}$) compared to the standard hydrogen electrode, low toxicity and easy synthesis, and sodium has an abundant provision from sea water [1-5].

The structure and operating principle of sodium-ion batteries (SIBs) and lithium-ion batteries (LIBs) are similar. However, the radius of sodium ion is larger than lithium

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ion, so the ability of sodium ion to intercalate into and extract from the structure of electrode materials is less than lithium ion. The radius large of Na^+ ions, which induces sluggish kinetics, large volume changes during sodiation-desodiation, a low capacity, and a short SIBs cycle life [4-7]. Therefore, to develop high-performance SIBs, suitable electrode materials, particularly anode materials with outstanding properties, should be explored. Many types of anodes have been developed by researchers, including sodium metal, oxide-based, carbon-based, and alloys of sodium metal suitable for anodes. In addition, the anode materials need to have a potential of less than 2.5 V vs. Na/Na^+ [8-19]. Table 1 below presents some of the anode materials that have been studied for sodium-ion batteries.

Table 1. Some anode electrodes studied for sodium-ion batteries

Electrode materials	Capacities (mAh.g^{-1})	References
Sodium metal	1166	[1]
Graphite	284	[7]
Hard carbon from cellulose	300	[7]
Soft carbon	270	[7]
Grapheme 3D foams	594	[7]
Na-Sb	660	[10-12]
Na-Sn	847	[13, 14]
Sb@NC	622.5	[17]
$\text{Na}_2\text{Ti}_3\text{O}_7$	189	[20]
$\text{Na}_2\text{Mo}_2\text{O}_7$	200	[21, 22]
MoO_3	410	[23]

Sodium metal has been studied by many researchers as the negative electrode in sodium-ion batteries, because it is a good anode for energy storage of sodium-ion batteries, its large capacity (1166 mAh.g^{-1}). However, the large reactivity of sodium metal with organic electrolyte solvents and the production of dendrites during Na metal deposition are even more troublesome, and the low melting point of Na (98°C) poses a considerable safety hazard in devices intended for operation at room temperatures [1].

The carbonaceous materials have excellent cyclic stability but suffer from insufficient specific capacity (from 200 mAh.g^{-1} to 594 mAh.g^{-1}) and extremely low potential plateaus ($\sim 0.1 \text{ V vs. Na}/\text{Na}^+$), which facilitates for Na dendrites growth, it causes short circuit and battery explosion [7].

Some alloys of Na with metal as Na-Sn, Na-Sb are a type of high-capacity anode materials. Alloy Na-Sn is one of the most intensively studied anode materials for SIBs

among the alloy-type anodes due to its low reaction potential and relatively high theoretical specific capacity of 847 mAh.g^{-1} . However, the volume expansion reaches 420% associated with the formation of $\text{Na}_{15}\text{Sn}_4$. The alloy Na-Sn electrode will quickly pulverize upon sodiation/desodiation reactions and consequently lose electrical contact with the current collector. Na-Sb alloy anodes also experience phenomena similar to those of Na-Sn alloy anodes [13, 14].

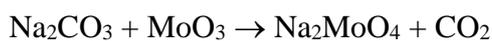
Another type of high-capacity anode materials for SIBS are transition metal compounds, including sulfides [24], selenides [25], transition-metal oxides, and sodium salts of transition metals because transition metals usually possess various oxidation states. One of among them have oxides of molybdenum, and molybdate compounds. The XRD data of all the molybdate compounds were indexed on the orthorhombic system. Therefore, the molybdate compounds has great potential as an anode for sodium-ion batteries [21-23, 26-28].

The crystalline structure of sodium molybdate (Na_2MoO_4) is the orthorhombic system, it is created by tetrahedral molybdate (MoO_4) arranged in layers. The crystal structure of Na_2MoO_4 has many similarities to the orthorhombic structure of MoO_3 [27, 28], which allows sodium ions to intercalate and separate easily. Therefore, the Na-Mo-O material system has great potential to form anodes for sodium ion batteries. In this study, Na_2MoO_4 material is reported as a promising anode for sodium ion batteries.

2. Experiments

2.1. Synthesis of NMO materials

A mixture of sodium carbonate (Na_2CO_3 , Sigma-Aldrich), (MoO_3 , Sigma-Aldrich), was fixed at a molar ratio such that $\text{Na}_2\text{CO}_3:\text{MoO}_3 = 1:1$. The mixture was carefully ground using mortar and pestle for 15 min, and then ball milled at 100 rpm for 1 h. The used balls were quartz with a diameter of 8mm, the weight ratio of balls and materials was: 20:1. The obtained homogeneous mixture was calcined at appropriate temperature through 2 steps: the mixed was first heat-treated at 500°C for 8 h in the air to remove carbonate in salts and then they were calcined at 800°C for 10 h to obtain the Na_2MoO_4 material. The reaction expected for Na_2MoO_4 was:



The synthesized Na_2MoO_4 materials, then it is determined morphological, structural and electrochemical properties.

2.2. Material characterizations

The crystal structure and morphology characteristics of Na_2MoO_4 were confirmed by X-ray diffraction (XRD, Siemens D5005 X-ray diffraction-meter with Cu-K α

radiation), scanning electron microscopy (SEM, JEOL JSM-6490), energy-dispersive X-ray spectroscopy (EDS) (SEM, JEOL JSM-6490).

The electrochemical characteristics of Na_2MoO_4 was evaluated through cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) on a Metrohm Autolab PGSTAT 302 N. The charge/discharge capacity at different current densities between 0.2 - 2.2 V was taken on a NEWARE battery testing system (BTS).

The electrochemical measurements are performed on CR2032-type sodium-ion battery, where cathode is the Na_2MoO_4 , anode is sodium foil, polypropylene (PE) membrane as separator and solution of 1 M NaClO_4 in a mixture of ethylene carbonate/diethylene carbonate (EC/DEC, 1:1 by volume) as the electrolyte. The CR2032 coin cells were assembled in a glove box with oxygen and moisture level less than 0.1 ppm and stabilized for 24 h before electrochemical performance testing. To prepare electrode materials, the slurry was formed by mixing active material (Na_2MoO_4), black carbon (supper P), and polyvinylidene fluoride (PVDF) as binder at weight ratio of 8:1:1 in N-methyl-2-pyrrolidone (NMP); then, the slurry was uniformly coated on a 15 μm -thick aluminum foil by tape coating, dried at 100°C in a vacuum oven for 12 h and cut into sheets ($\text{Na}_2\text{MoO}_4\text{@C}$).

3. Results and discussions

3.1. Structural and morphological characterization

The phase structure of synthesized Na_2MoO_4 materials and Na_2MoO_4 mixtures with black carbon to prepare electrode and charged to 2 V ($\text{Na}_2\text{MoO}_4\text{@C}$) are depicted in Fig. 1.

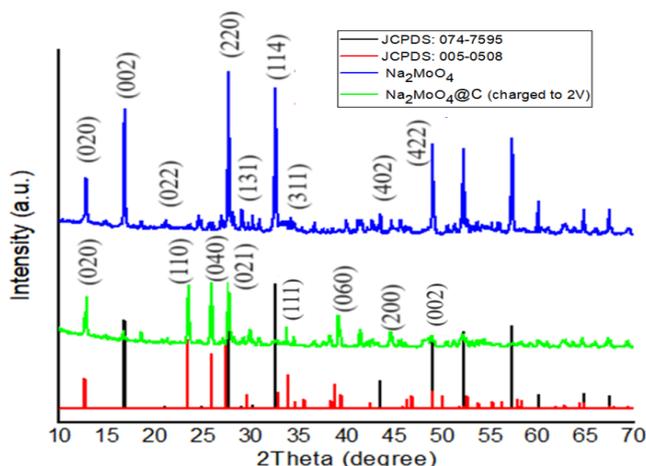


Fig. 1. XRD pattern of Na_2MoO_4 , and $\text{Na}_2\text{MoO}_4\text{@C}$ (charged to 2V) materials.

As can be seen in Fig. 1, the XRD pattern of synthesized Na_2MoO_4 material has diffraction peaks observed at 12.79°, 16.83°, 23.54°, 27.65°, 30.19°, 32.55°, 34.19°, 45.52°, and 49.10°. These diffraction peaks correspond to planes properly identified

which are (020), (002), (022), (220), (131), (114), (311), (402), and (422), respectively. In comparison with the JCPDS card No 074-7595 standard, the diffraction peaks on XRD pattern of synthesized Na_2MoO_4 material has a corresponding coincidence at the peaks. The Na_2MoO_4 material have orthorhombic structure. Na^{1+} is bonded to six equivalent O^{2-} atoms to form NaO_6 octahedra that share corners with six equivalent MoO_4 tetrahedra and edges with six equivalent NaO_6 octahedra. Mo^{6+} is bonded to four equivalent O^{2-} atoms to form MoO_4 tetrahedra that share corners with twelve equivalent NaO_6 octahedral [27, 28]. In addition, on the diffraction spectrum of the synthesized material, there are other diffraction peaks that coincide with the JCDPS card No #05-0508 standard of $\alpha\text{-MoO}_3$. It shows that the synthesized material still has MoO_3 which has not completely reacted.

Also presented in Fig. 1, the XRD of $\text{Na}_2\text{MoO}_4\text{@C}$ material with diffraction peaks observed at 12.82° , 23.45° , 25.94° , 27.43° , 33.72° , 39.19° , 44.65° , and 48.92° are properly identified to the (020), (110), (040), (021), (111), (060), (200), and (002), planes, respectively. When Compared to the JCPDS #05-0508 standard of $\alpha\text{-MoO}_3$, the diffraction peaks on XRD pattern of $\text{Na}_2\text{MoO}_4\text{@C}$ observed is quite similar to the diffraction spectrum of orthorhombic $\alpha\text{-MoO}_3$. The crystal structure of $\alpha\text{-MoO}_3$ ($\alpha\text{-MoO}_{11}\text{O}_{22}\text{O}_{33}$) has a unique two dimensional layer structure in which each layer is built up of MoO_6 octahedrons connected along ac-planes by common edges and corners to form zigzag rows and along ab-planes by common corners only. The interlayer interaction is weak and bounded in the a axis direction by van der Waals forces [24]. With a stacked layer structure, Na_2MoO_4 and $\text{Na}_2\text{MoO}_4\text{@C}$ materials are facilitated the intercalation and separation of sodium ions during the charge-discharge process, and it is a potential candidate for the anode of sodium-ion batteries.

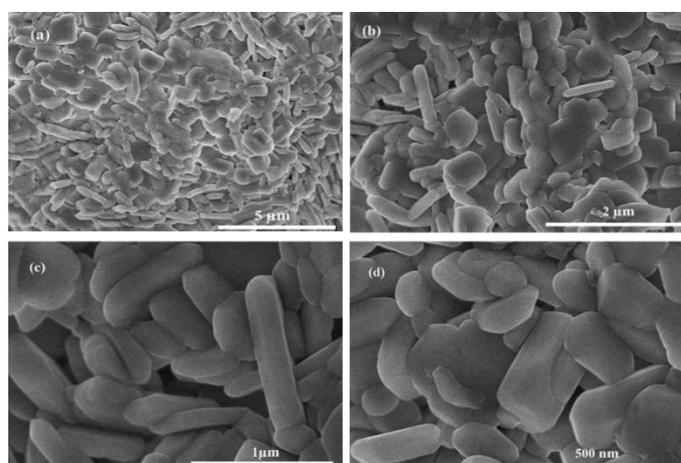


Fig. 2. SEM images of Na_2MoO_4 material at different magnifications; (a) ten thousand times; (b) twenty thousand times; (c) fifty thousand times; (d) one hundred thousand times.

The composition of the Na_2MoO_4 materials was evaluated by energy dispersive X-ray spectroscopy (EDS) method, as shown in Fig. 3. The result presented that the synthesized material contained Na, Mo, and O elements. The atomic ratio between the Na, Mo, and O elements is approximate 2:1:4, which is consistent with the initially used raw precursor ratio.

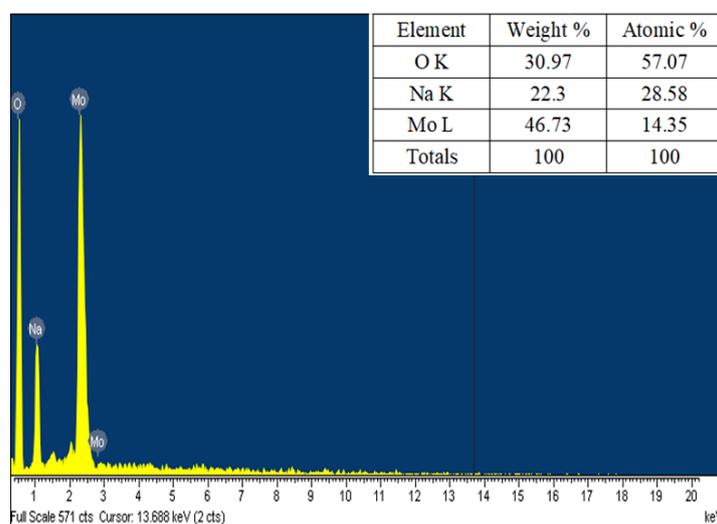


Fig. 3. Energy dispersive X-ray spectroscopy (EDS) result of Na_2MoO_4 .

3.2. Electrochemical properties

Figure 4a is charge/discharge profiles of the Na_2MoO_4 material at the current density of 0.1 C (40 mA.g^{-1}) with voltage range from 0.2 to 2.2 V. The first charge curves and discharge have created a relatively flat voltage plateau in the voltage range from 0.5 to 0.8V. This shows a superiority of Na_2MoO_4 material. The first charge capacity and discharge capacity are approximately 430 mAh.g^{-1} and 420 mAh.g^{-1} , respectively. The charge-discharge voltage is slowly changed in the voltage range of 0.5-0.8 V that means the largest contribution of capacity in the charge-discharge process occurred between 0.5 to 0.8 V vs. Na/Na^+ . The superiority of Na_2MoO_4 materials has shown that they have great potential as anodes for sodium-ion batteries.

Figure 4b shows the charge-discharge capacity for 50 cycles of the Na_2MoO_4 material at a current density of 0.1 C. The specific capacity of the material is gradually decreased to approximately 325 mAh.g^{-1} at the 50th cycle. The capacity remains about 80% of its first cycle. The cyclic efficiency of the synthesized material is the same some reported anode materials [7, 21, 22].

Figure 4c and Fig. 4d illustrate the charge-discharge curves and specific capacity of Na_2MoO_4 material at current densities of 0.1 C, 0.2 C, 0.5 C, and 1 C. The capacity decreases significantly from 420 mAh.g^{-1} at 0.1 C (from the 1st to 3rd cycle) to

230 mAh.g⁻¹ at 1 C (from the 10th to 12th cycle). When the current density is backed to 0.1 C (from the 13th to 15th cycle) the capacity regained 410 mAh.g⁻¹, which means the structure of Na₂MoO₄ material is stable after cycling at high current densities. The material has high capacity when charge/discharge at low current density and it also remains quite high capacity when charge/discharge at high current density. In other words, the internal resistance of the material is slightly increased when increases the charging-discharging current density. The charging and discharging at high current densities, the voltage increases moderately during charging and decreases marginally during discharging compared to charging and discharging at low current densities (between 0.4 V and 0.8 V) (Fig. 4c).

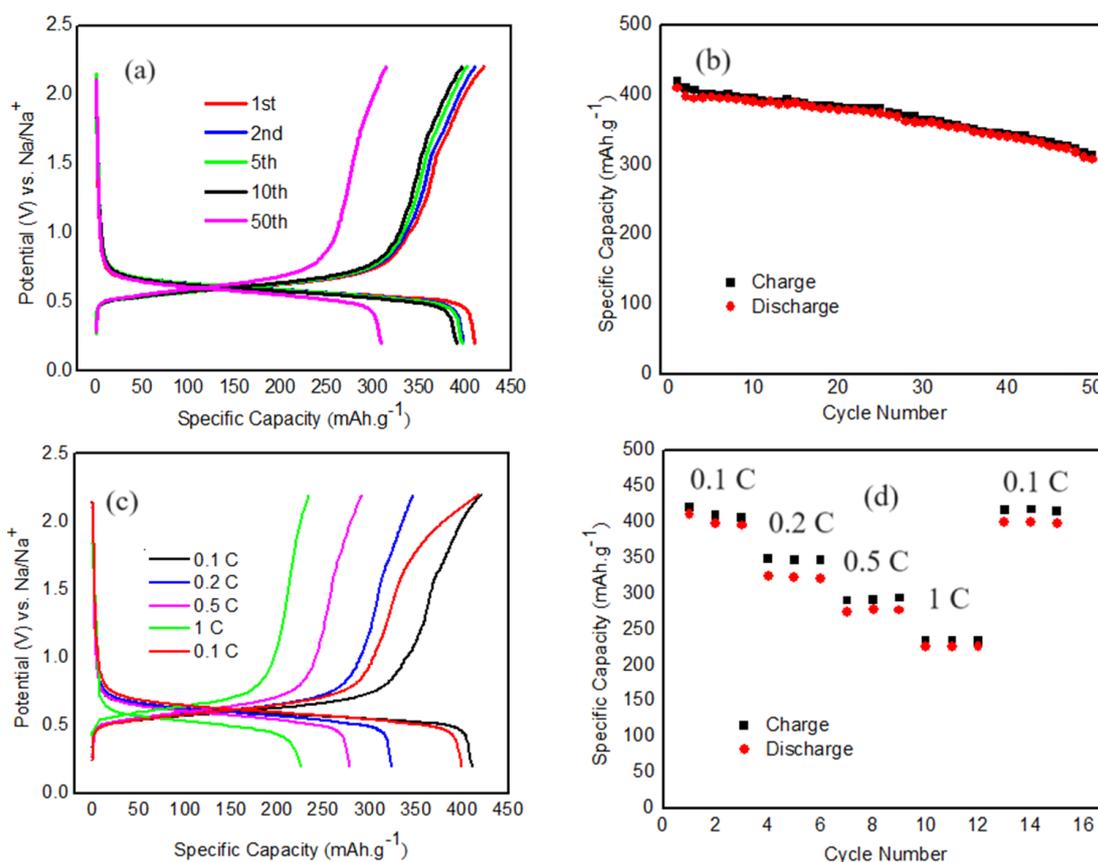


Fig. 4. (a, b) The charging-discharging profiles of NMO at 0.1 C; (c, d) The charging-discharging profiles at different current densities.

The cyclic voltammetry (CV) scanning of the Na₂MoO₄ material is shown in Fig. 5a. The CV scanning was carried out in the range from 0.2 to 2.5 V with a scanning speed of 0.1 mV.s⁻¹ on Metrohm Autolab PGSTAT 302 N, with the working electrode connected to the positive terminal of the battery, counter and reference electrodes are connected to the negative terminal of the battery.

On the CV spectrum, there is a pair of peaks, which correspond to the voltage plateaus in the charge-discharge processes. The pair of peaks appeared at about 0.5 V and 1.2 V, which correspond to the insertion process and the extraction process of Na^+ ion on Na_2MoO_4 electrode material, respectively. The area of the peaks is relatively large, which it has proved the Na_2MoO_4 material that have high specific capacities.

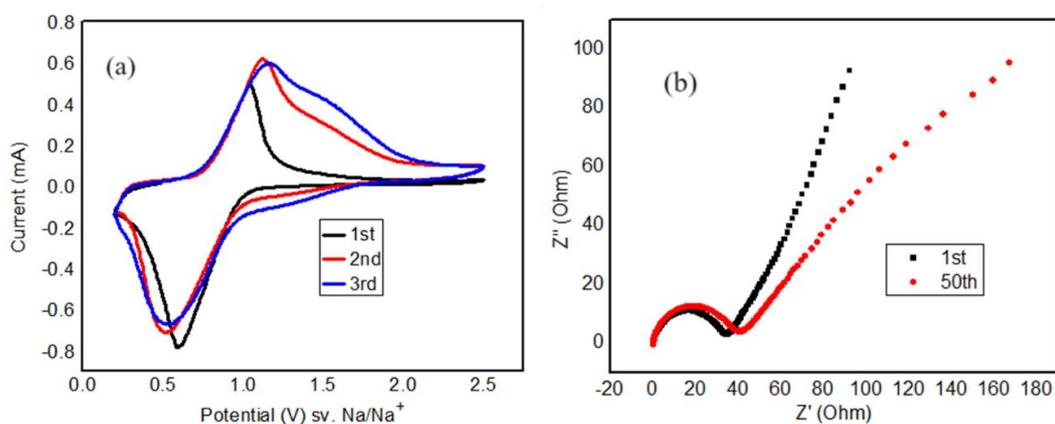


Fig. 5. (a) The CV curves of Na_2MoO_4 at 0.1 mV s^{-1} ; (b) Nyquist plots of the Na_2MoO_4 materials.

The electrochemical impedance spectroscopy (EIS) characterization was conducted with various frequencies ranging from 1 MHz to 0.1 Hz to evaluate the electrochemical reaction kinetics, as shown in Fig. 5b. The Nyquist plots of Na_2MoO_4 material at the 1st and 50th cycles are similar. They have a semicircle at high frequency and an inclined line at low frequency. The total impedance is also relatively low. It proves that the Na_2MoO_4 material is quite stable during the reversible intercalation and extraction of Na^+ ions into the material structure.

4. Conclusions

The layered structure of sodium molybdate Na_2MoO_4 materials was successfully synthesized by a conventional solid-state reaction method. The structure of Na_2MoO_4 materials is the similar orthorhombic structure of Na_2MoO_4 (JCPDS #074-7595). When are charged to 2 V vs. Na/Na^+ , the structure of Na_2MoO_4 is the same of orthorhombic $\alpha\text{-MoO}_3$ (JCPDS #05-0508). The SEM and EDS results confirmed Na_2MoO_4 materials has particle size from about 300 nm to 1 μm and it has the approximate combinatorial formula: Na_2MoO_4 . The electrochemical properties of Na_2MoO_4 materials is evaluated by cyclic voltammetry (CV), charging-discharging with constant current density, and electrochemical impedance spectroscopy (EIS). The Na_2MoO_4 material has a high specific capacity of 420 mAh.g^{-1} at the first cycle. The capacity remains about 325 mAh.g^{-1} after 50 cycles. The results suggest that the Na_2MoO_4 material is a potential anode material for sodium-ion batteries.

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CỰC ÂM NĂNG LƯỢNG CAO CHO PIN NATRI-ION DỰA TRÊN VẬT LIỆU NATRI MOLYBDAT

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Tóm tắt: Trong nghiên cứu này, natri molybdat (Na₂MoO₄) được tổng hợp bằng phương pháp phản ứng ở trạng thái rắn. Cấu trúc tinh thể và hình thái của vật liệu natri molybdat (Na₂MoO₄) đã được nghiên cứu bằng phương pháp nhiễu xạ tia X (XRD), kính hiển vi điện tử quét (SEM) và quang phổ tia X phân tán năng lượng (EDS). Vật liệu Na₂MoO₄ được sử dụng để chế tạo pin đồng xu loại CR2032 và sau đó được đánh giá về các đặc tính điện hóa. Các đặc tính điện hóa của Na₂MoO₄ được đánh giá thông qua phép đo quyết thể vòng tuần hoàn (CV), phổ tổng trở điện hóa (EIS) và phóng-nạp ở các mật độ dòng điện khác nhau trên hệ thống kiểm tra pin NEWARE (BTS). Kết quả nghiên cứu tính chất điện hóa của vật liệu Na₂MoO₄ cho thấy nó có tính chất điện hóa khá tốt. Đặc biệt, vật liệu điện cực Na₂MoO₄ đã đạt được dung lượng riêng vượt trội vào khoảng 420 mAh.g⁻¹ ở 0,1 C trong dải điện áp từ 0,2 đến 2,2 V. Vật liệu Na₂MoO₄ thể hiện hiệu suất chu kỳ khá tốt, có khả năng duy trì tới 80% dung lượng riêng trong 50 chu kỳ ở mật độ dòng điện là 0,1 C. Ngoài ra, khi sạc/xả ở mật độ dòng điện cao 1 C, Na₂MoO₄ giữ được dung lượng riêng khoảng 240 mAh.g⁻¹. Kết quả cho thấy vật liệu Na₂MoO₄ là cực âm đầy hứa hẹn cho pin natri-ion.

Từ khóa: Pin natri-ion; natri molybdat; vật liệu anode; phản ứng ở trạng thái rắn.

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