

CeVO₄-coated LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ as positive material: towards the excellent electrochemical performance at normal and high temperature

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Abstract

The CeVO₄-coated LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ (NCM 622) cathode materials are successfully synthesized by hydrothermal method. The structure, morphology and electrochemical properties of the surface modified NCM 622 materials are characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and constant current charge and discharge test. The SEM images and XPS patterns show that nanosized CeVO₄ layer is uniformly coated on the surface of NCM 622 active material. Furthermore, the electrochemical performance of all the CeVO₄-coated NCM 622 samples are improved significantly. 3 wt% of CeVO₄-coated NCM 622 cathode material exhibits specific capacity of 146.1 mAh g-1 and excellent capacity retention (89.63%) between 2.8 and 4.3 V after 100 cycles at elevated temperature (60 °C). The alternating current impedance and cyclic voltammetry tests show that the CeVO₄ coating can reduce the electrode polarization and enhance the electrochemical activity of cathode materials.

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1 Introduction

Lithium-ion batteries (LIBs) as the important new energy device are widely used in the electric vehicles (EVs), hybrid electric vehicles (HEVs) and other appliances [1-3]. In the field of lithium-ion batteries, cathode materials are widely studied. As the most common lithium ion battery cathode material, LiCoO₂ is difficult to meet the development of cathode material for lithium ion battery because of its high cost, high toxicity and low capacity [4, 5]. While LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ (NCM 622) is a kind of layered $LiNi_{x}Co_{y}Mn_{1-x-y}O_{2}$ material is considered as one of the most promising commercial cathode materials with impressive specific capacity, high operating voltage and environment friendliness [6]. However, the layered NCM 622 has poor cycling performance at high temperatures and high cutoff voltages because those two factors accelerate the harmful side reactions between the active material and the electrolyte and finally destroy the surface configuration of the material [7]. In order to solve such a problem, what is usually done is to protect the surface of the cathode material from the electrolyte. In some literature, partial substitution and coating modification of active material have proven to be effective measures to improve electrochemical stability [8–11]. In the above method, the surface modification by the various of cladding materials such as ZrO₂ [12], LiAlO₂ [13], TiO₂

[14], $Li_2Si_2O_5$ [15], $Mn_3(PO_4)_2$ [16] have been reported to improve the electrochemical performance of NCM 622. The coating layer not only reduces the side reaction between the active material and the electrolyte, but also forms a better solid electrolyte layer (SEI film) on the cathode surface [17, 18]. Chen et al. [19] synthesized Al₂O₃-coated NCM 622 cathode materials by ultrasonic method, which improved the capacity retention of NCM 622 from 89 to 93.9% after 30 cycles. Furthermore, Lee et al. [20] applied Li_3PO_4 to the surface of NCM 622 material via a citric acid-assisted sol-gel method to enhance the cycling performance at high cut-off voltages (the capacity retention of 79.7% after 100 cycles at 1 C under a high charge cut-off voltage of 4.7 V). All of the above results confirm the positive effects of coating layer on the NCM 622 cathode materials for lithium-ion batteries.

As a kind of rare earth compound, $CeVO_4$ exhibits good resistance to both acid corrosion and oxidation at elevated temperatures and can also be used as an impressive Li⁺ conducting medium. Zhang et al. [21] prepared a $CeVO_4$ electrode material with good electrochemical performance by a hydrothermal method and the discharge capacity was still 210 mAh g⁻¹ after 50 cycles (capacity retention about 75%). In this work, a facile hydrothermal method was used to coat NCM 622 with $CeVO_4$ as a raw material for the first time. Furthermore, the structure and morphology of all the samples are systematically analyzed to explore the impact of the $CeVO_4$ modification. The electrochemical performance of modified NCM 622 at both normal and elevated temperature are also been investigated.

2 Experimental

2.1 Preparation

The Ni_{0.6}Co_{0.2}Mn_{0.2}(OH)₂ precursor was produced via a co-precipitation method. NiSO₄·6H₂O, CoSO₄·7H₂O and MnSO₄·H₂O were mixed in deionized water (n(Ni): n(Co): n(Mn) = 6:2:2). The reaction temperature was kept at 60 °C and pH was adjusted by NaOH solution to 10–11. And then, a mixture of dehydrated Ni_{0.6}Co_{0.2}Mn_{0.2}(OH)₂ and Li_2CO_3 (n(Li): n(Ni_{0.6}Co_{0.2}Mn_{0.2}(OH)₂) = 1.05:1) sintered at 600 °C for 6 h and then at 900 °C for 7 h to form $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$ material.

The CeVO₄ coated materials were prepared by hydrothermal method. Ce(NO₃)₃·7H₂O and Na₃VO₄·12H₂O were dissolved in deionized water (n(Ce(NO₃)₃·7H₂O): n(Na₃VO₄·12H₂O) = 1:1). After that, the suspension was transferred into Teflon-lined autoclave and kept under 180 °C for 24 h. The resulting precipitate was washed and dried with deionized water. Then absolute ethanol for 12 h to obtain CeVO₄ material.

In order to obtain a CeVO₄-coated NCM 622 cathode materials. NCM 622 was dissolved in CeVO₄ solution and the suspension was transferred into a Teflon-lined autoclave and kept at 180 °C for 24 h and calcined at 500 °C for 5 h to obtain CeVO₄-modified NCM 622 material. The prepared samples contain different contents of CeVO₄ and the expected weight ratios of CeVO₄ to NCM 622 are 1.0, 3.0, and 5.0 wt% (labeled as 1.0-NCM 622, 3.0-NCM 622 and 5.0-NCM 622, respectively). The whole synthesis process was shown in Fig. 1.

2.2 Characterization

The crystal structure of the powders was identified by powder X-ray diffraction (XRD, D/MAX-2200 X) using Cu K α radiation in the 2 θ range of 10°–80° at a continuous scan mode with a step size of 0.02° and a scan rate of 10° min⁻¹. The surface morphology of the bare and coated materials was observed by measurement of scanning electron microscopy method (SEM, JEOL JSM-5600LV). The element distribution of samples was characterized by energy dispersive spectrometer method (EDS). The chemical valence state was analyzed by X-ray photoelectron spectroscopy (XPS, PHI 5600, Perkine-Elmer, USA) employing an Al K α excitation source.

2.3 Electrochemical measurements

The cathode materials assembled into CR2032 button cell to carry out electrochemical performance test. Button cell are made in an argon-filled glove box, the cathode material



consists of 80 wt% of coated active materials, 10 wt% of conductive acetylene black and 10 wt% of polyvinylidene fluoride (PVDF) binder dissolved in *N*-methyl-2-pyrrolidone (NMP) solvent. Lithium metal foil was used as anode, the electrolyte is consisted of a solution of 1 M LiPF₆ in ethyl-ene carbonate (EC), dimethyl carbonate (DMC) and ethyl methyl carbonate (EMC) (1:1:1 in volume). The charge/discharge properties were tested by CT-3008 battery test system (Shenzhen Newware Electronics, Ltd.) between 2.8 and 4.3 V (vs. Li/Li⁺). Moreover, the test of cyclic voltammetry curve (CV) and electrochemical impedance spectroscopy (EIS) was carried out on an electrochemical workstation (CHI 720B, China).

3 Results and discussion

The XRD patterns of the pristine and modified samples are displayed in Fig. 2. The diffraction peaks are assigned to the (003), (101), (006), (102), (104), (105), (107), (108), (110), and (113) planes of NCM622 (JPCDS No. 09-0063), and no extra diffraction peaks are evident. All the samples have a layered hexagonal α -NaFeO₂ structure belonging to the R3m space group [22]. The apparent division of the (108)/(110) and (006)/(102) peaks indicates that all the samples have a good layered structure [9]. No peak associated with CeVO₄ is observed even when the coating amount is up to 5 wt%, which may be due to the less coating amount [23]. It can be seen form Table 1 that the lattice constant of LiNi_{0.6}Co_{0.2}Mn_{0.2}O₂ is similar to that reported in previous literature [24]. The intensity ratio of I_{003}/I_{104} (R) is always considered as an indication of cation mixing. With the increased ratio of I_{003}/I_{104} , the disorder degree of the material becomes alleviating. Generally, when the ratio is



Fig. 2 XRD patterns of the pristine and CeVO₄-coated samples

 Table 1
 Refined lattice parameters of pristine and CeVO₄-coated

 NCM 622 samples
 \$\$^2\$

Sample	a/(Å)	c/(Å)	c/a	I ₍₀₀₃₎ /I ₍₁₀₄₎
The pristine	2.869	14.242	4.9641	1.348
1.0-NCM 622	2.868	14.240	4.9651	1.348
3.0-NCM 622	2.869	14.241	4.9637	1.351
5.0-NCM 622	2.870	14.241	4.9620	1.349

higher than 1.2, the materials have a good layered structure with small cation mixing [25]. In this work, all the samples have a high intensity ratio of I_{003}/I_{104} (nearly 1.35), indicating good layered structures with low cation mixing of NCM 622 samples before and after coating.

The SEM images of the pristine and CeVO₄-coated NCM 622 samples (1.0-NCM 622, 3.0-NCM 622, 5.0-NCM 622) are presented in Fig. 3. As seen from the SEM images, all of particles are well spheroid with particle size of $10-20 \mu m$. Furthermore, compared with the bare one, there are some small particles attached to the surface of CeVO₄ modified samples as well as the coating on NCM 622 particles becomes intensive with the increase of coating amount. The analysis implies that the successful coating of CeVO₄ could be observed and the CeVO₄ coating layer does not affect the morphology of the active material.

The composition and distribution of elements on the surface of 5.0-NCM 622 sample are examined by energy dispersive spectrometer method (EDS). The EDS mapping (Fig. 4) results show that the elements (Ce and V) are distributed uniformly on the surface of particles of $CeVO_4$ -coated NCM 622 samples. It is clear from the figure that $CeVO_4$ particles is successfully covered on the surface of NCM 622.

Figure 5 shows the XPS spectra of the pristine and 3.0-NCM 622 samples. It is noteworthy that the peaks at binding energies of 885.7 and 905.9 eV are correspond to Ce3d_{5/2} and $\text{Ce3d}_{3/2}$, and then the peaks at binding energies of 516.9 and 524.6 eV are assigned to $V2p_{3/2}$ and $V2p_{1/2}$, which indicates that the Ce and V on the material surface of 3.0-NCM 622 are Ce³⁺ and V⁵⁺, respectively [26, 27]. The electron binding energies of Co2p_{3/2} and Mn2p_{3/2} appear at 779.3 and 642.7 eV, which indicate that Co and Mn are presented as Co³⁺ and Mn⁴⁺, respectively. Furthermore, the peaks with binding energies of 854.6 eV is assigned to Ni $2p_{3/2}$ peak, which suggest that the Ni element maintain at Ni²⁺ [28]. The Ni 2p, Co 2p, and Mn 2p peaks of 3.0-NCM 622 are not significantly shifted compared to the pristine NCM 622 samples, corresponding to the peaks of Ni²⁺, Co³⁺, and Mn⁴⁺, respectively, indicating that CeVO₄ cladding not affect the Crystal structure of NCM 622 material. It is noted that the peaks of Mn 2p, Co 2p and Ni 2p are significantly decreased after coating, while the O 1 s peak of the 5.0-NCM 622 is significantly higher than the bare one, indicating there exists





Fig. 4 The SEM patterns of 5.0-NCM 622 (**a**) and the EDS mappings of 5.0-NCM 622 (**b–d**)



Fig. 5 XPS spectra of the pristine and 3.0-NCM 622 samples. a Ni 2p; b Co 2p; c Mn 2p; d O 1s and e Ce 3d; f V 2p

much more oxygen on the surface of 3.0-NCM 622 material due to the presence of $CeVO_4$ [29, 30].

To explore the effect of $CeVO_4$ coating on the electrochemical performance of the modified material, the electrochemical performance of the pristine and $CeVO_4$ -coated samples is tested in the voltage range of 2.8–4.3 V at room temperature. Figure 6a indicates the initial charge/discharge profiles of the pristine and $CeVO_4$ -coated samples. All the curves show the voltage platform of NCM 622 samples around 3.8 V, which corresponds to the redox reaction of Ni^{2+}/Ni^{4+} [31]. The initial charge/discharge capacities of the pristine NCM 622 sample are 193.1 and 169.9 mAh g⁻¹, respectively, with corresponding coulomb efficiency of 87.99%. While the coulomb efficiencies of 1.0-NCM 622, 3.0-NCM 622 and 5.0-NCM 622 are 90.17, 91.11, and 90.54%, respectively. Furthermore, the discharge capacity of CeVO₄-coated NCM 622 samples are slightly lower than that of the pristine NCM 622 sample, because CeVO₄



Fig. 6 a Initial charge/discharge curves of pristine and CeVO₄-coated samples at 0.1 C. b Rate performance of pristine and CeVO₄-coated samples at different current density

surface coating reduces the proportion of active material [32].

Figure 6b exhibits the rate capabilities of the pristine and $CeVO_4$ -coated samples under different current density. The capacity of all samples decreases with the increase of the current density. It is noted that the $CeVO_4$ -coated NCM 622 samples exhibits a favorable rate capability than the bare one. For instance, the discharge capacity of 3.0-NCM 622 sample is 105.10 mAh g⁻¹ at 5 C, retaining 64.09% of the original 164.0 mAh g⁻¹ at 0.1 C, respectively. While the pristine sample delivers only 91.41 mAh g⁻¹ at 5 C, retaining 53.79% of the original 169.9 mAh g⁻¹ at 0.1 C, respectively. The result indicates that the rate capability of NCM 622 samples are enhanced by $CeVO_4$ coating, which may be due to $CeVO_4$ coating can not only effectively avoid side

reactions between the active material and the electrolyte, but also promote the solid electrolyte interface (SEI) film formed on the surface of the active material [19].

Figure 7 illustrates the cycling performance of the pristine and CeVO₄-coated samples at 1 C rate between 2.8 and 4.3 V at 25 and 60 °C. Obviously, the capacities of all the samples retrograde gradually with the increase of the cycle number. Furthermore, CeVO₄-coated samples exhibit better cycling performance than the bare one at 25 and 60 °C. As can be seen from Fig. 7a, after 100 cycles, the corresponding discharge capacity of pristine NCM 622 sample is remain 124.5 mAh g⁻¹, which the corresponding capacity retentions of 79.95%. The capacity retention of CeVO₄-coated NCM 622 samples are 89.89, 92.68, and 91.19%, respectively. The result shows that CeVO₄-coated NCM 622 has higher



Fig. 7 a Cycling performance of the pristine and CeVO₄-coated samples at 25 °C; b cycling performance of the pristine and CeVO₄-coated samples at 60 °C at 1 C

cycling performance, with a 3.0 wt% CeVO₄ coating showing the best cycling performance. The result indicates that CeVO₄ coating layer could obviously improve the cycling performance of the cathode materials. The reason for the increased electrochemical performance may be due to the CeVO₄ coating inhibiting the side reaction between the electrode and the electrolyte caused by HF etching [33].

Figure 7b shows the cycling performances of the pristine and CeVO₄-coated NCM 622 samples between 2.8 and 4.3 V at high temperature (60 °C). It is noted that the initial discharge capacities of the pristine and CeVO₄-coated at 60 °C are higher than the cycling capacity at 25 °C. This phenomenon is probably related to the improved diffusion rate of Li⁺ at high temperatures. After 100 cycles, the corresponding discharge capacity of pristine NCM 622 sample is remain 103.9 mAh g⁻¹, which the corresponding capacity retentions of 65.89%. The capacity retention of CeVO₄-coated NCM 622 samples are 88.20, 89.63, and 87.69%, respectively. All the CeVO₄-coated NCM 622 samples exhibit a higher cycling performance than the bare one. Apparently,

 Table 2
 The comparison with the cycling performance of similar positive materials

Coating materials of NCM-622	The capacity retention after several cycles at certain rate under different cutoff volt- ages		
CeVO ₄ (our work)	92.68% 100 cycles, 1 C, 4.3 V		
Al ₂ O ₃ [19]	95%, 30 cycles, 0.2 C, 4.5 V		
Mn ₃ (PO ₄) ₂ [16]	93.3%, 50 cycles, 0.5 C, 4.3 V		
SiO ₂ [25]	93%, 50 cycles, 1 C, 4.3 V		
Li ₂ ZrO ₃ [39]	85%, 50 cycles, 0.1 C, 4.8 V		
Li ₂ Si ₂ O ₅ [15]	67%, 200 cycles, 5 C, 4.5 V		
Li ₃ PO ₄ [20]	79.7%, 100 cycles, 1 C, 4.7 V		

the 3 wt% CeVO₄ coating is the most effective in improving the cycling performance of NCM 622. Obviously, the CeVO₄-coated samples are shows higher capacity retentions and better cycling stability even at high temperature.

To investigate the effect of $CeVO_4$ on the performance of NCM 622, the cycling performance of $CeVO_4$ -coated NCM 622 cathode material and other similar cathode materials was compared. The relevant comparison data is shown in the Table 2. It is noted that the $CeVO_4$ -coated NCM 622 cathode material exhibit good capcitiy retentions compared to other similar cathode materials. It is proved that the coating modification of $CeVO_4$ is an effective method to improve the cycling performance of NCM 622 cathode material.

Figure 8 shows the cyclic voltammogram (CV) of the pristine and 3.0-NCM 622 samples between 2.8 and 4.3 V after 1st, 50th and 100th cycles at a scan rate of 0.1 mV/s. It can be seen from Fig. 8 that all the samples have only one pair of redox peaks at 2.8–4.3 V, which corresponding to the Ni²⁺/Ni⁴⁺ redox reaction [34, 35]. The gaps (Δ V) between the oxidation peak and the reduction peak reflects the electrochemical reversibility of the electrode [36]. The Δ V of 3.0-NCM 622 sample after 1st, 50th and 100th cycles are 0.161, 0.129 and 0.137, respectively, which is lower than uncoated one (0.193, 0.168 and 0.166). The results indicate that the CeVO₄-coated sample has better electrochemical reversibility and lower polarization, which is consistent with the results of the cycling performance tests discussed above.

To further investigate the change in electrochemical properties after CeVO_4 coating, electrochemical impedance spectroscopy (EIS) tests are performed for the pristine and 3.0-NCM 622 samples after 20th and 50th cycles between 2.8 and 4.3 V. The impedance spectra of the pristine and 3.0-NCM 622 samples after different cycles as well as the equivalent circuit model are showed in Fig. 9.



Fig. 8 Cyclic voltammograms (CV) of a the pristine and b 3.0-NCM 622 samples between 2.8 and 4.3 V



Fig. 9 Nyquist curves of a the pristine and b 3.0-NCM 622 after 20th, and 50th at 25 °C

Table 3Impedence data of pristine and 3.0-NCM 622 after differentnumber of cycles at equilibrium state

Cycle number	The pristine		3.0-NCM 622	
	Rs/ohm	Rct/ohm	Rs/ohm	Rct/ohm
20th	3.941	87.54	3.870	58.41
50th	4.680	192.7	4.644	103.2

Obviously, all EIS curves consist of a depressed semicircle in the high to medium frequency and the oblique line in the low frequency. The semicircle from the high frequency to the mid-frequency region represents the interfacial charge transfer resistance (Rct), and the oblique line represents Warburg impedance (W₀) which is related to the impedance of Li⁺ diffusion [37]. The fitting parameters are listed in Table 3, all EIS are fitted by the software Zview 2.0 (shown in Fig. 9c). It is clearly observed that the Rs values of the pristine and 3.0-NCM 622 samples have no significant change, while the CeVO₄-coated NCM 622 samples exhibits the lower Rct values than the bare one. The possible explanation is that the $CeVO_4$ coating layer effectively inhibits the side reaction between the active material and the electrolyte, thereby enhancing the diffusion efficiency of Li⁺. Therefore, the CeVO₄ coating layer effectively reduces the impedance of the active material [38, 39], which is consistent with the results of cycling and rate performances shown in Figs. 4 and 5.

4 Conclusions

In this work, CeVO₄-coated NCM 622 materials are successfully prepared via hydrothermal method. The CeVO₄ coating has no obvious influence on the crystalline structure of the substrate. Furthermore, the coating suppresses the directly contacting between the active materials and electrolyte and improve the resistance to elevated temperatures of active materials. As a result, the CeVO₄-coated NCM 622 samples exhibited better electrochemical performance both normal and elevated temperatures. Typically, 3 wt% of CeVO₄-coated NCM 622 exhibits specific capacity of 146.2 mAh g⁻¹ and excellent capacity retention (90.76%) between 2.8 and 4.3 V after 100 cycles at high temperature (60 °C). In addition, EIS measurements clearly show that the CeVO₄ coating can effectively reduce the Rct of the cathode material. Therefore, we believe that surface modification by CeVO₄ is an effective method to ameliorate the electrochemical performance of NCM positive materials, and then this achievement could be extended to other electrodes to obtain favorable performance.

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