TECHNICAL ARTICLE





Improving Electrochemical Performance of NMC622 Cathode by Coating with Cr₂O₃ Nanopowders and Modified Current Collector

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Submitted: 9 March 2022 / Revised: 20 August 2022 / Accepted: 25 September 2022

The surface of $\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$ particles and current collector was modified with Cr_2O_3 , and NaOH solution, respectively, and the electrochemical performance of NMC622 as a cathode material of Li-ion battery was investigated. The contact angle test shows the more wettability of the modified Al foil, confirming the better adhesion of cathode material slurry on that. The cell with a modified current collector has more capacity (176.6 mAh g⁻¹ at 0.1C) at the first cycle and better cyclic performance (capacity loss of 12.8% after 50 cycles) than the cell with the unmodified current collector (172.3 mAh g⁻¹ at 0.1C, and capacity loss of 17% after 50 cycles). Moreover, modifying the surface of NMC622 particles as a cathode active material with 0.5 wt.% of Cr_2O_3 and surface treatment of the current collector leads to a high capacity (179.8 mAh g⁻¹ at 0.1C) and improved cyclic stability; capacity loss of 9.1% after 50 cycles.

Keywords	cathode,	Li-ion	battery,	modified	current	collector,
	surface coating, X-ray diffraction					

1. Introduction

The demand for renewable energy storage is highly increased, because of the related problems with fossil fuels. In order to solve the problems caused by fossil fuels, energy storage systems such as lithium-ion batteries and recently, sodium-ion batteries have been considered.

Li-ion batteries have an essential role in developing portable electronic productions. The use of Li-ion batteries has been remarkable in the progress of electric vehicles due to their high energy density and power density (Ref 1-3). All parts of the Li-ion and Na-ion batteries are impressive in achieving better quality and high performance. Nevertheless, the breakthrough of large–scale energy storage systems will significantly depend on developing of high-performance cathode materials (Ref 4-6).

 $LiCoO_2$ is the first generation of commercial cathode materials in Li-ion batteries, which has an intensive interest due to its high practical voltage, high capacity, and good rate capability. Because of high cost and the toxicity of cobalt (Ref 7), nickel-rich materials with layer structure (LiNi_xM_{1-x}O₂, M=Co, Mn, and Al; x > 0.5) have been considered as a proper candidate for cathode materials (Ref 4, 8, 9). However, the high content of nickel brings about structural instability. This instability is ascribed to the dissolution of transition metal ions into electrolyte that causes the formation of an inactive solid electrolyte interface (CEI) on the cathode surface (Ref 10, 11). For decreasing the undesired reactions, the surface coating of cathode active materials has been investigated. According to the presented reports, this process protects the cathode electrode against side reactions with HF acid and reduces oxygen loss from cathode material [12]. Recently, synthesis and investigation of the electrochemical performance of Li-Ni_{0.6}Mn_{0.2}Co_{0.2}O₂ (NMC666) have been carried out as the promising material for the cathode of Li-ion battery (Ref [13-15]). Furthermore, to improve its cyclic stability, surface coating by metal oxides such as SiO₂ (Ref 16), ZrO₂ (Ref 8), and TiO_2 (Ref 17) has been investigated.

 Cr_2O_3 has also been reported as a surface modifier for cathode materials of $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$ (Ref 18) and $LiMn_2O_4$ (Ref 19). The obtained results show increasing the electrochemical cyclic performance.

The slurry of cathode material, binder, and carbon is coated on the Al foil as a current collector. The current collector is an effective component during the charge and discharge cycles. Current collectors must have good mechanical, chemical and electrochemical stability. Current collectors provide the pathways for conducting the electrons to the active layers that are supported. Therefore, good contact between the active layer and the current collector reduces the interface resistance, which must be maintained during the charge and discharge cycles (Ref 20). The surface modification of Al foil is suggested for enhancing the adhesion of cathode material to the current collector. There are a few reports on the surface coating of Al foil. Carbon black/graphene on the modified aluminum foil as a current collector showed an improvement in both cyclic performance and rate capability (Ref 21). The use of conducting polymer, as a modifier material for Al foils is also reported

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(Ref 22). Moreover, the synthesis of a thin compact oxide layer on Al foil via the oxidation process indicated the increased adhesion of active material to the current collector (Ref 23). Furthermore, there are some reports about chemical etching, indicating the improvement of electrochemical performance (Ref 24, 25).

In this work, we examined an easy method to increase the adhesion of NMC622 to the Al foil surface and also modified the surface of NMC622 particles with Cr_2O_3 to investigate their effects on the electrochemical performance.

2. Experimental section

Commercial powder of NMC622, LiPF₆ solution, PVDF, NMP, conductive carbon (super P), Al foil, lithium chip, and separator (Celgard 2300) were purchased from Xiamen TOB New Energy Technology Co., NaOH pellets from Merck and Cr_2O_3 nanopowders from US-NANO.

The current collector was modified through the following procedure. First, the surface of Al foil was well cleaned with a normal detergent solution. The foil was washed with water and dried, and then cleaned with acetone. In the second step, the Al foil was scrubbed with an aqueous solution of NaOH for about 1 min. After washing with deionized water, the foils dried in the oven at 80 °C for 30 min. The modified and unmodified Al foils were referred as M-Al and P-Al, and the weight of their cut foils with a diameter of 15 mm was 9 mg and 9.7 mg, respectively. For modification of NMC622 particles, Cr₂O₃ and NMC622 powders were mixed in ethanol by a focused ultrasonic wave. The weight percentages of Cr₂O₃ were 0.5 and 1 wt.%. The produced mixture was dried at 60 °C for four hours on a magnetic stirrer, followed by four hours heating, at 500 °C in an air atmosphere with a heating and cooling rate of 5 °C/min.

To determine of the NaOH effect on wettability of Al foil, contact angle measurement was carried out by the method of sessile drop with an accuracy of 0.1° . (Adeeco, Iran).

The pristine NMC622 and the coated one with Cr_2O_3 were investigated by structural and morphological analyses; x-ray diffraction (XRD, GNR-EXPLORER, Italy), scanning electron microscopy (SEM, VP 1450; LEO–Germany), and transition electron microscopy (TEM, AB-912; LEO– Germany).

To examine the modification effect of Al foil on electrochemical performance, the prepared electrodes with M-Al and P-Al foils were fabricated. For this, a slurry of active material (NMC622), carbon black, and polyvinylidene fluoride binder (PVDF) with the weight percentage of 94:3:3, respectively, was coated on the foils. The coating thickness was about 30 μ m. Nmethyl-2-pyrrolidone (NMP) was used as a solvent. The outcome product dried at 120 °C for twelve hours, in a vacuum oven. To examine the prepared electrodes, a coin cell configuration (CR2032) was selected and assembled in a glove box filled with argon. Coin cells consisted of prepared electrodes (working electrode), Li chip (as the reference electrode), 1 M $LiPF_6$ (electrolyte), and separator (Celgard 2300). The assembled cells with pristine NMC622 deposited on Al foil with and without surface treatment were labeled as M-Al and P-Al, respectively. The cells containing modified NMC622 powders with Cr₂O₃ coated on the M-Al foil were also made with a similar process and named M-Al-Cr₂O₃ (0.5 wt.%) and M-Al-Cr₂O₃ (1 wt.%).

Galvanostatic charge–discharge tests were performed by a battery test instrument (BTS4000-5V10mA). Cyclic voltammetry and electrochemical impedance spectroscopy measurements of fabricated cells were carried out by Origaflex-OGF 01 instruments.

3. Result and Discussion

3.1 Surface Wettability Investigation

The contact angle measurement was carried out by using NMP as a liquid on the surfaces of the pristine Al foil (P-Al foil) and modified Al foil (M-Al foil). Their contact angles were determined by a contact angle goniometer device. The contact angle of NMP on the M-Al foil (27.8 $^{\circ}\pm$ 0.1°) was smaller than that of the P-Al foil (46.9 \pm° 0.1°). The better wetting of modified Al foil can increase the adhesion of cathode slurry on the current collector due to the roughness created on the Al foil. This phenomenon can be related to the corrosive effect of NaOH solution. In this process, the scrubbing time was set at 1 min, while shorter times had no significant effect and longer times caused foil destruction.

3.2 Phase Analysis

Figure 1 and 2 shows the XRD patterns and the Rietveld refinement patterns of the commercial NMC622 powders and modified powders with 0.5 wt.% and 1 wt.% of Cr_2O_3 . Our investigations showed that the coating percentage of 2% and 3% caused a worse electrochemical performance than that of 0.5 wt.% and 1 wt.%. So all subsequent investigations were performed using 0.5 wt.% and 1wt.% coating.

All patterns confirm the presence of characteristic peaks related to the layered structure of hexagonal α -NaFeO₂ (space group: R-3 m, No. 166) without any impurity phases. The absence of a diffraction pattern of Cr₂O₃ is due to the low concentration of Cr₂O₃ coating. There is not any significant shift in the peaks for modified powders relative to pristine powders, as shown on the right side of Fig. 1. The values of refined lattice parameters for all samples are presented in Table 1. The samples coated with Cr₂O₃ do not show any considerable effect on the cell dimension of NMC622. However, there is a decrease in the intensity of peaks for the



Fig. 1 XRD patterns of NMC622 powders, pristine and modified with 0.5 and 1 wt.% of Cr_2O_3



Fig. 2 Rietveld refinement patterns of NMC622 powders, pristine and modified with 0.5 and 1 wt.% of Cr_2O_3

sample with 1 wt.% of Cr_2O_3 . It illustrates the crystallinity reduction in NMC622 powders modified by 1 wt.% Cr_2O_3 .

3.3 Microstructure of Pristine and Cr₂O₃-Coated Powders

Figure 3 presents the SEM images of pristine powders and modified powders with 0.5 and 1wt.% of Cr_2O_3 . The shape of particles is almost spherical, consisting of irregular polyhedral tiny particles in all cases. By increasing the percentage of

 Table 1
 Refined lattice parameters of all samples

Samples	a, Å	c, Å	R _{wp}	Chi ²	
Pristine	2.867	14.218	13.2	3.1	
0.5 wt.%	2.869	14.219	7.4	3.3	
1 wt.%	2.868	14.217	7.3	1.6	

 Cr_2O_3 to 1 wt.%, the tiny particles, are more agglomerated. Therefore, their crystallinity is decreased, which is confirmed by x-ray diffraction data.

TEM analysis was conducted to identify the coated Cr_2O_3 layer visually on the surface of NCM622particles. For an example, Fig. 4 shows TEM images of the pristine powder and a sample with 0.5 wt.% Cr_2O_3 . According to Fig. 4a, there is no extra coated layer on the particles of pristine NMC622. Whereas, Fig. 4b confirms the presence of a thin layer of Cr_2O_3 particles with a thickness lower than 20 nm on the particle surface of 0.5 wt.% Cr_2O_3 -NMC622.

3.4 Electrochemical Performance

Figure 5 shows the initial charge–discharge curves of the cells with the different specifications at 0.1C. The discharge specific capacity of the P-Al cell was 172.3 mAh g^{-1} , which is lower than that of the M-Al cell (176.6 mAh g^{-1}).

Moreover, the discharge specific capacities of modified NMC622 with 0.5 and 1 wt.% of Cr_2O_3 were 179.8 and 178.9 mAh g⁻¹, respectively. According to these results, treating the current collector and coating of the NMC622 powders are effective methods to achieve a better discharge capacity.

Figure 6 illustrates the cyclic voltammetry curves of the cells after the first cycle in the voltage range of 2.8-4.3 V with a scan rate of 0.1 mV s⁻¹. Each curve contains two peaks related to the redox reaction of Ni^{2+}/Ni^{4+} during the lithiation/ delitiation process [8].

The voltage difference between anodic and cathodic peaks determines the reversibility of reduction/oxidation reactions (Table 2).

Among all the cells, the modified NMC622 with 0.5 wt.% of Cr_2O_3 has the lowest voltage separation and better electrochemical reversibility due to decreased polarization. Moreover, the intensity of current density peaks is maximum for the M-Al- Cr_2O_3 (0.5 wt.%) sample, which is in agreement with the data of initial charge–discharge curves (Fig. 5).

Figure 7 illustrates the cyclic performance of the cells at a rate of 1C for 50 cycles. The calculated coulombic efficiency of all samples is above 99% after 2th cycle, while the sample of M-Al-Cr₂O₃ (0.5 wt.%) has the largest initial coulombic efficiency. The data extracted from Fig. 7 are presented in Table 3. The cell made from M-Al has a lower capacity loss (12.8%) than that of the cell with P-Al (17%). The cell composed of particles with 0.5 wt.% Cr₂O₃ coated on the M-Al decreases the capacity loss to 9.1%.

Furthermore, the values of discharge capacity of the M-Al-Cr₂O₃ (0.5 wt.%) sample at 1 C after the first and 50th cycles are higher (156.8 and 142.6 mAh g⁻¹, respectively) than that of other samples. M-Al-Cr₂O₃ (1 wt.%) sample has the maximum capacity loss. This result can be assigned to the decrease in crystallinity of the particles in this sample which is also confirmed by XRD patterns and SEM images.



Fig. 3 SEM images of NMC622 powders, pristine and modified with 0.5 and 1 wt.% of $\rm Cr_2O_3$



Fig. 4 TEM images of NMC powders, (a) pristine and (b) modified with 0.5 wt.% of Cr₂O₃



Fig. 5 Initial charge–discharge curves of all samples at the rate of 0.1C



Fig. 6 Cyclic voltammetry curves of samples (a) P-Al, (b) M-Al, (c) M-Al- Cr_2O_3 (0.5 wt.%), and (d) M-Al- Cr_2O_3 (1 wt.%)

Table 2Electrochemical parameters obtained from CVcurves of the samples

	Potential values, V				
Sample	V _{pc}	$V_{ m pa}$	$\Delta V_{\rm p}$		
P-Al	3.790	3.688	0.102		
M-Al	3.801	3.706	0.095		
M-Al-Cr ₂ O ₃ (0.5 wt.%)	3.779	3.692	0.087		
M-Al-Cr ₂ O ₃ (1 wt.%)	3.801	3.684	0.117		

The adhesion strength is an essential factor for having a good and stable contact between the cathode material and the current collector, which leads to the mechanical stability of the electrode (Ref 23). Besides, the CEI layer created between cathode active material and electrolyte prevents the fast diffusion of Li^+ and brings about capacity reduction.



Fig. 7 Cyclic performance of the produced samples at 1C

Our data show that the surface treatment of the Al current collector and the surface modification of NMC622 particles with a low amount of Cr_2O_3 are two effective agents to improve the cyclic performance and the amount of delivered discharge capacity.

According to presented reports about the effect of Cr_2O_3 coating on $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ and $LiMn_2O_4$ particles, a determined percentage of Cr_2O_3 coating can decrease the harmful electrochemical reaction and provide a better performance (Ref 18, 19) which supports our outcome data.

Woosuk Cho et al. (Ref 16) reported that SiO₂ coating can improve the electrochemical performance of commercial NMC622. After 50 cycles, the capacity of pure NMC622 was 158.1 mAh g⁻¹ with a capacity retention of 94%, and capacities of NMC622 coated by SiO₂ was 159.5, 158.3, 156.4 mAh g⁻¹ for 0.5%, 1%, and 3%, respectively, with retention capacities of 95%, 95.4%, and 97%.

Woosuk Cho et al. (Ref 10) investigated the coating effect of $Mn_3(PO_4)_2$ on commercial NMC622 powders. The capacity of uncoated NMC622 was 153 mAh g^{-1} after 50 cycles corresponding to a retention capacity of 92.6%, while NMC622 coated with $Mn_3(PO_4)_2$ showed the capacity of 149 mAh g^{-1} with a more capacity retention of 93.3%. So, the $Mn_3(PO_4)_2$ coating slightly improved the cycling performance of commercial powders.

TiO2 coating on commercial NMC622 was reported by Qin et al. (Ref 17) According to the results, TiO₂@NCM-622 showed a discharge capacity of TiO₂@NCM-622 of 185.6 mAh g^{-1} at 1C with capacity retention of 80.8% after 100 cycles, about 20% higher than that of the bare sample.

Electrochemical impedance spectroscopy analysis was measured after the first charge–discharge cycle from 100 to 15 mHz with 10 mV amplitude. EIS measurements were carried out to study the modification effect of Al surface and NMC particles on the electrochemical kinetics of the electrodes. Figure 8 presents Nyquist plots of all samples, displaying two semicircles followed by an inclined line. The intersection with the Z_{real} axis in the high-frequency region represents the internal ohmic resistance (R_s). The semicircle in the high frequency is ascribed to the CEI film resistance (R_f). The semicircle in the low frequency is attributed to the charge transfer resistance (R_{ct}) between the interface of electrode and electrolyte. The inclined line in the low-frequency region is related to Li-ion diffusion through the active material particles (Ref 26). As shown, R_s is

 Table 3 Discharge capacity at 1C and capacity loss of the samples

	Discharge mAh	e capacity, g ⁻¹		
Sample	First cycle 50		Capacity loss, %	
P-A1	139.2	115.5	17	
M-Al	144.9	126.3	12.8	
M-Al-Cr ₂ O ₃ (0.5 wt.%) M-Al-Cr ₂ O ₃ (1 wt.%)	156.8 153.2	142.6 122.2	9.1 20.2	



Fig. 8 EIS spectra of the assembled cells after first cycle. The inset indicates the equivalent circuit for fitting

Table 4 Fitting result of the equivalent circuit from Nyquist curves and calculation result of Li^+ diffusion coefficient for the samples

Samples	$R_{\rm s}, \Omega$	$R_{\rm f}, \Omega$	R_{ct}, Ω	$\sigma_{ m w}$	${\rm D_{Li}}^+$, cm s ⁻¹
P-A1	7.621	39.29	367.8	13.706	0.95×10^{-12}
M-Al	3.335	14.63	308.6	12.781	1.09×10^{-12}
M-Al-Cr ₂ O ₃ (0.5 wt.%)	5.661	9.181	216.9	9.939	1.80×10^{-12}
M-Al-Cr ₂ O ₃ (1 wt.%)	6.519	10.41	451.8	14.665	0.83×10^{-12}

almost the same for all cells. The M-Al cell has a smaller R_f than that of the P-Al cell. Surface treatment of Al foil causes better contact between the NMC622 particles and the Al foil, preventing the formation of a surface layer on the particles (Ref 27). Moreover, Cr₂O₃ coating on the NMC622 particles in M-Al-Cr₂O₃ (0.5 wt.%) and M-Al-Cr₂O₃ (1 wt.%) cells brings about a more decrease in R_f relative to uncoated particles in M-Al cell, as observed from the diameter of semicircles in the high frequency.

The diameter of the semicircle in the low frequency is lower for the M-Al-Cr₂O₃ (0.5 wt.%) cell, illustrating lower charge transfer resistance (R_{ct}) of this sample than that of the other samples. Coating the NMC622 particles with 0.5 wt.%-Cr₂O₃



Fig. 9 Rate capability performance of (a) M-Al and (b) M-Al- Cr_2O_3 (0.5 wt.%) samples

reduces the side reactions between electrode and electrolyte and increases the ionic conductivity. Despite the positive effect of Cr_2O_3 coating on the reduction of side reactions, agglomeration of coated NMC622 particles with 1 wt.%- Cr_2O_3 leads to a bigger R_{ct} and a decrease of ionic conductivity. The fitting results of the equivalent circuit from Nyquist curves for the samples are shown in Table 4.

Moreover, the diffusion coefficient of Li ions (D_{Li^+} was calculated using EIS analysis by the following formula:

$$D_{Li^+} = \frac{R^2 T^2}{2A^2 n^2 F^4 C^2 \cdot \sigma_w^2}$$
(Eq 1)

In this formula, R is the gas constant (8.314 J mol⁻¹ K⁻¹), T is the absolute temperature (298 K), A is the surface area of the working electrode (2 cm²), n is the number of electrons per molecule during oxidization (n = 1), F is Faraday constant (96,485 C mol⁻¹), C is Li-ion concentration (0.00718 mol cm⁻³), and σ_w is the Warburg coefficient, the curve slope of $Z_{real} \sim w^{-1/2}$ [28]. As presented in Table 4, the achieved results show that the sample of M-Al-Cr₂O₃ (0.5 wt.%) has a higher diffusion and improved Li⁺ kinetics than other samples, verifying CV analysis.

The rate capability performance of M-Al and M-Al-Cr₂O₃ (0.5 wt.%) samples is illustrated in Fig. 9. The charge– discharge process was carried out at the rates of 0.1C, 0.5C, 1 C, 2C, 5C, 10C, and then 0.1C. In all rates, M-Al-Cr₂O₃ (0.5 wt.%) sample delivers more discharge capacity. This result can be attributed to increasing the ionic conductivity and decreasing the polarization by modifying the surface of the NMC622 with Cr₂O₃, which is compatible with the result of the EIS analysis.

4. Conclusions

The commercial NMC622 was chosen as cathode material to investigate the effects of surface treatment of Al foil and coated NMC622 with Cr_2O_3 on its electrochemical performance.

The surface of Al foil was modified by NaOH solution, used as a current collector in Li-ion cells. Contact angle measurement shows that the surface treatment brings about the better wettability of the Al foils and increases the contact between foil and cathode material. The investigation of electrochemical measurements shows that the cathode active material on the treated Al foil has greater cyclic stability than that of unmodified Al foil. Also, Al foil treatment and the surface modification of NMC622 particles with the suitable amount of Cr_2O_3 (0.5 wt.%) is examined. The outcome results indicate a decrease in polarization and charge transfer resistance, leading to a greater specific capacity, capacity retention, and rate capability. The enhanced electrochemical performance of the M-Al- Cr_2O_3 (0.5 wt.%) electrode is due to the decrease in side reactions between cathode material and electrolyte.

Acknowledgments

The authors thank the Ferdowsi University of Mashhad for their support.

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