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Washing effect on properties of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ cathode material by ethanol solvent

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Abstract: Different $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ cathode materials were washed by ethanol solvent. Inductively coupled plasma atomic emission spectroscopy (ICP-AES), Fourier transformed infrared (FTIR) spectrum, X-ray diffraction (XRD), scanning electron microscopy (SEM), charge−discharge test and electrochemical impedance spectroscopy (EIS) were used to evaluate the elemental contents, structures, morphologies and electrochemical properties of samples. The results show that ethanol washing can remove effectively the synthetic residues $LiOH/Li_2O$ on the freshly-prepared $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ and make the sample much more resistant to H_2O and CO_2, without destroying its bulk structure, surface morphology and electrochemical performances. Moreover, the discharge specific capacity and cycle performance of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ after storage in air with a relative humidity of 80% for three months are improved by immediate ethanol washing.

Key words: lithium-ion battery; $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$; ethanol washing; storage property; electrochemical performance

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1 Introduction

Layered Ni-rich materials $LiNi_{1-x-y}Co_xM_yO_2$ ($0.8\le 1-x-y<1$, M=Al, Mn, Mg, etc) have attracted much attention for power sources due to their large capacity, excellent rate capability and low cost [1−5]. However, their storage property has been considered to be very poor on account of the formation of Li_2CO_3 on the surface [6−9]. Li_2CO_3 is electrochemically inactive and easily causes gas evolution during the battery operation [6].

It is not denied that lithium ions in Li_2CO_3 derive from the as-prepared materials. During the preparation process of Ni-rich materials, excess $LiOH\cdot H_2O$ or other lithium compounds were added. Unreacted lithium compounds present in the form of $LiOH/Li_2O$ on the fresh powder account for the very rapid appearance of Li_2CO_3 on the surface of the fresh material exposed to air. Thus, two approaches have been used to remove the lithium residues on the particle surface. One is to coat the material with oxides or phosphates which can react with lithium residues to form new compounds, such as $LiVO_2/LiV_2O_5$ [10,11], $Li_2O-2B_2O_3$ [12], Li_xCoPO_4 [13], $LiMnPO_4$ [14], $LiCoO_2$ [15], FeF_3 [16] and Li_2TiO_3 [17]. The storage performance and electrochemical properties of $LiNi_{1-x-y}Co_xM_yO_2$ materials can be enhanced due to the elimination of $LiOH/Li_2CO_3$ and the formation of the lithiated compounds on its surface. The other is to wash the material by water to remove $LiOH/Li_2CO_3$ owing to its solubility in water [7,8,18]. However, this method needs subsequent heat treatment to recover the electrochemical properties of the material because the washing process can create more NiO-like cubic phase [7].

In this work, a facile process is reported for the elimination of lithium residues based on the different solubility characteristics of LiOH and Li_2CO_3 in ethanol solvent, which is expected to enhance the storage performance of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ without destroying its bulk structure and surface morphology.

2 Experimental

$LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ was synthesized from $Ni_{0.8}Co_{0.15}Al_{0.05}OOH$ according to our previous report [1].

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The freshly-prepared powders were marked as "F" and used for further experiments. At 25 °C, 20 g F-powders were dispersed in 50 mL ethanol under constant stirring with a magnetic stirrer for 15 min. The powders recovered by filtration were dried at 150 °C. This procedure was repeated 1−3 times. Correspondingly, the powders treated with ethanol 1−3 times were marked as "EF*n*" (*n*=1, 2 or 3), respectively. In addition, 20 g $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ powders placed in air for 1 d underwent the same treatment as the freshly-prepared powders. And the obtained samples were marked as "ES*n*" (*n*=1, 2 or 3), respectively. According to our previous investigation [15], the condition of 80% relative humidity was the most terrible for the storage property of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, which led to the rapid decrease of the discharge capacity of the material. Therefore, in this work, the storage process was carried out at room temperature by storing the sample in air with a relative humidity of 80% for three months. The mass increasing rate (Δm) and average nickel oxidation state (OS) of the sample were calculated according to our previous report [15].

The contents of lithium, nickel, cobalt and aluminum of the samples were measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES, Optima 4300DV). The moisture content (OH^-) was determined by a Karl−Fisher moisture titrator at 250 °C. The carbon content in Li_2CO_3 was measured by a CHS element analyzer at 1000 °C in an oxygen atmosphere. Fourier transformed infrared (FTIR) spectrum was tested on Shimadzu-FTIR−8400 spectrometer. The morphology of the sample was observed by a scanning electron microscopy (SEM, SU 3500). The structure of the sample was characterized by a powder X-ray diffraction (XRD, D/max-r A type, Cu K_{α}).

CR2032 coin-type cells were assembled with metallic lithium anodes for electrochemical tests. The positive electrode consisted of 80% active material, 10% acetylene black and 10% PVDF binder (mass fraction). The cells were assembled in an argon filled glove box. The charge−discharge measurement was performed at a current density of 0.2*C* within the voltage range of 2.8−4.3 V (vs Li/Li^+) at room temperature on a LAND CT2001A test system. Electrochemical impedance spectroscopy (EIS) experiments were performed at full discharge state of desired cycle using a CHI660B electrochemical workstation over the frequency range of $1\times10^{5}-1\times10^{-3}$ Hz with the amplitude of ±5 mV.

3 Results and discussion

3.1 Changes of chemical composition, oxidation state, moisture and carbon contents

Table 1 gives the chemical composition of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ before and after being washed in ethanol solvent. It is obvious that the lithium content of sample F is the highest owing to the addition of excess $LiOH\cdot H_2O$ in its preparation. Once sample F is washed in ethanol solvent for 1−3 times, the contents of lithium, nickel, cobalt and aluminum are close to theoretical values, as shown in sample EF1, EF2 and EF3. However, after sample F placed in air for 1 d undergoes the same treatment, the contents of all elements almost does not change, as shown in sample ES1, ES2 and ES3. This may be attributed to the terrible storage condition of 80% relative humidity in air, which leads to the rapid reaction between residual $LiOH/Li_2O$ and CO_2 to form Li_2CO_3 [7]. It is well-known that LiOH can be dissolved by appropriate amount of ethanol, while Li_2CO_3 is insoluble in ethanol. Therefore, ethanol washing is only used for the freshly-prepared $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$.

Table 1 also presents the nickel oxidation state (OS), moisture and carbon content in sample F, EF2 and ES2 before and after storage in air for three months. Before storage, OS values of sample F and EF2 are 3.000 and the carbon contents are also close, while the moisture of

Table 1 Chemical compositions of samples F, EF*n* and ES*n* (*n*=1, 2 or 3), nickel oxidation state (OS), moisture and carbon contents in samples F, EF2 and ES2 before and after storage in air for three months

Samlple	Chemical composition (mass fraction)/%				Oxidation state		Moisture content/10^{-6}		Carbon content/10^{-6}	
	Li	Ni	Co	Al	Before storage	After storage	Before storage	After storage	Before storage	After storage
F	7.29	48.83	9.16	1.38	3.000	2.916	611	3826	540	4142
EF1	7.23	48.87	9.17	1.39						
EF2	7.22	48.88	9.17	1.39	3.000	2.982	102	279	527	595
EF3	7.22	48.88	9.17	1.39						
ES1	7.28	48.84	9.16	1.38						
ES2	7.28	48.84	9.16	1.38	2.988	2.917	598	3810	563	4125
ES3	7.28	48.84	9.16	1.38						

sample EF2 is only one-sixth that of sample F, which indicates that ethonal washing can remove effectively residual LiOH on the fresh $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ without structural degradation. After storage, for sample F, OS decreases to 2.916, and moisture and carbon contents increase by about 6 and 7 times, respectively. However, sample EF2 shows a few changes. On the other hand, compared with sample F, sample ES2 shows the similar changes in the aspects of OS, moisture and carbon contents. These results indicate that during the course of storage, a certain amount of LiOH, Li_2CO_3 and NiO are formed on the surfaces of the fresh and placed powders. However, the removal of residues on the surface of the freshly-prepared powder can effectively reduce the forming amounts of LiOH, Li_2CO_3 and NiO. This may also show that the synthetic residue is the main source of deteriorating the storage property of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ and should be eliminated in time.

3.2 SEM and XRD studies

Figure 1 depicts the SEM images of samples F, EF2 and ES2. It is obvious that the surface morphology of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ is not affected by ethanol washing. Samples F, EF2 and ES2 all have spherical morphology in secondary particles and the estimated particle size is about 10 μm in diameter, while the primary particles are cylinder-shaped and they are densely agglomerated in secondary forms.

XRD patterns of samples F, EF2 and ES2 are shown in Fig. 2. It is observed that the three samples fit well with α-$NaFeO_2$ structure with no minor phases [1]. Distinct splitting of the (006)/(102) and (108)/(110) peaks is seen in these patterns. The obtained lattice parameters are presented in Fig. 2. Compared with sample F, the lattice parameters of the EF2 and ES2 show no obvious changes. This indicates that the samples possess typical layered characteristics before and after ethanol washing, and the washing process does not change the structure of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$.

3.3 Change of mass

Figure 3 shows the mass increasing rate curves of samples F, EF2 and ES2 during the course of storage. In the first 10 d, the masses of three samples all show increasing trend. However, the mass of sample F and ES2 increases more rapidly than that of sample EF2. After two weeks, the masses of samples F, EF2 and ES2 all stabilize and increase by 1.433%, 0.479% and 1.329%, respectively. These results indicate that ethanol washing can effectively enhance the storage property of fresh $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, but it is useless for the stale powders.

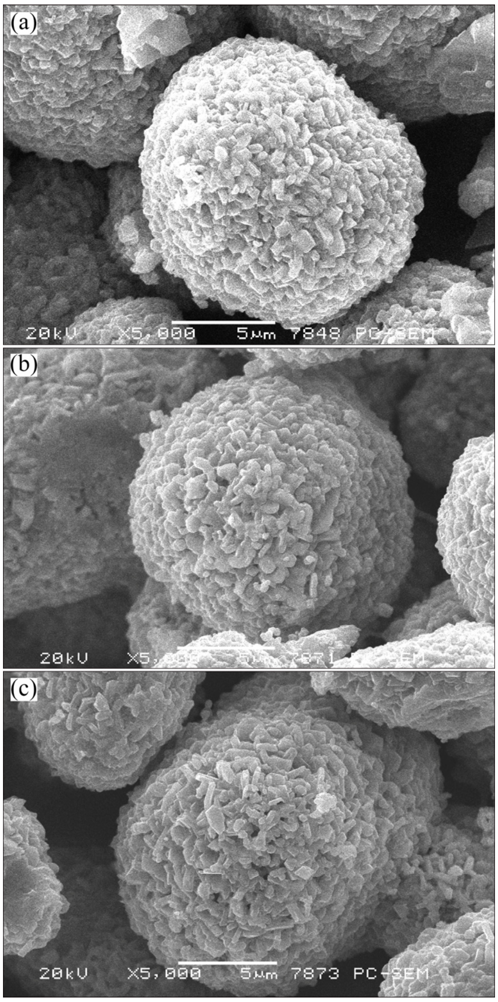

Fig. 1 SEM images of samples F (a), EF2 (b) and ES2 (c)

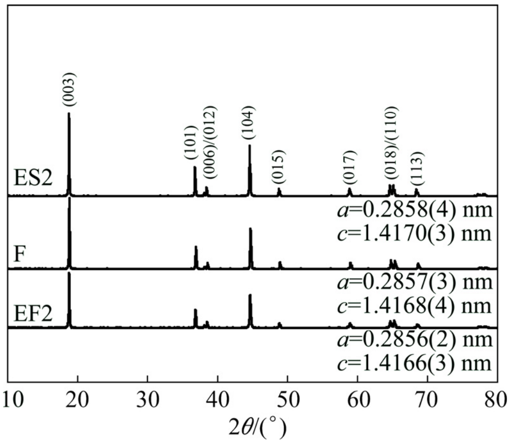

Fig. 2 XRD patterns of samples F, EF2 and ES2

3.4 FTIR study

Figure 4 shows FTIR spectra of fresh sample F, stored samples F, EF2 and ES2. Compared with fresh sample F, the FTIR spectra of the stored samples show significantly increased peaks of LiOH and Li_2CO_3. The band at 3600−3000 cm^{-1} is assigned to the O—H stretching vibration from LiOH, and two peaks at

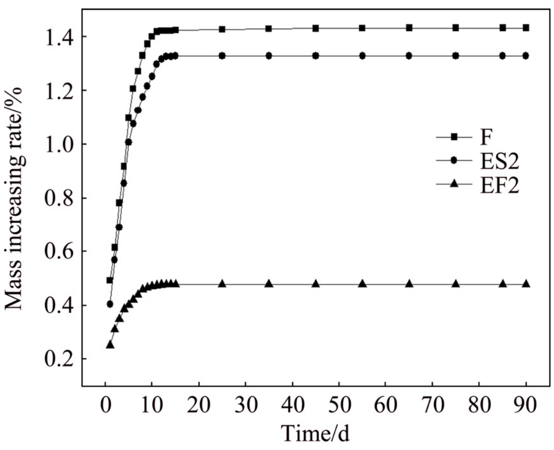

Fig. 3 Mass increasing rate curves of samples F, EF2 and ES2

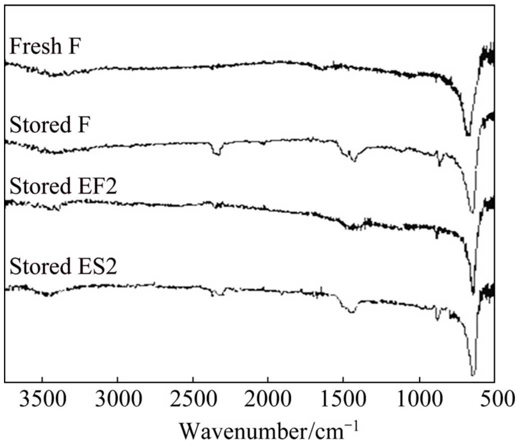

Fig. 4 FTIR spectra of fresh sample F, stored samples F, EF2 and ES2

868 and 1430 cm^{-1} are assigned to the CO_3^{2-} vibration from Li_2CO_3 [15]. The peaks of these bands for the stored sample F and stored ES2 are more prominent than those of the stored sample EF2. This means that the amounts of LiOH and Li_2CO_3 on the surface of sample EF2 are less than those of sample F and ES2 after exposure to air. Therefore, it is proven again that the removal of residual LiOH/Li_2O with ethanol washing can effectively reduce the forming amounts of LiOH/Li_2CO_3 on the surface of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ during the storage.

3.5 Analysis of discharge capacity

Figure 5 compares the first and thirtieth discharge curves of sample F, EF2 and ES2 before and after storage in air for 90 d. As shown in Fig. 5, for the freshly-synthesized sample F, the first and thirtieth discharge specific capacities are 196.8 and 190.9 mA·h/g, respectively, with the capacity retention of 97.0% after 30 cycles. However, after storage in air for 90 d, its first, thirtieth discharge specific capacities and capacity retention decrease to 127.5 mA·h/g, 84.8 mA·h/g and 66.5%, respectively. Contrastively, it is found from Fig. 5 that the fresh sample EF2 has almost the same electrochemical properties as the fresh sample F, but the stored sample EF2 has higher discharge capacity and better cycle performance than the stored sample F. After storage for 90 d, the stored sample EF2 has still the initial discharge capacity of 158.6 mA·h/g and the capacity retention of 82.1% after 30 cycles. On the other hand, as shown in Fig. 5, the fresh sample ES2 displays inferior electrochemical performance to the fresh sample F and EF2. Its initial discharge capacity and capacity retention after 30 cycles are 185.5 mA·h/g and 90.5%,

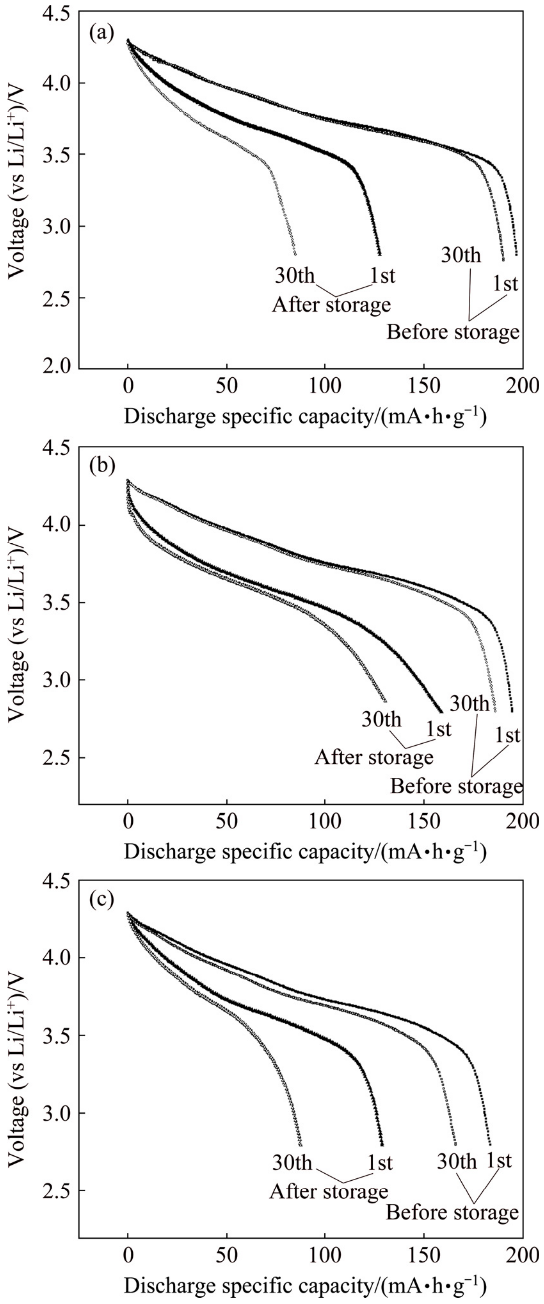

Fig. 5 First and thirtieth discharge curves of samples F (a), EF2 (b) and ES2 (c) before and after storage in air for three months

respectively. In addition, compared with the stored sample F, electrochemical properties of the stored sample ES2 are not improved at all.

3.6 EIS analysis

In order to elucidate the effect of ethanol washing on the electrochemical properties of the material, EIS experiments were performed for samples F, EF2 and ES2 before and after storage in air with a relative humidity of 80% for three months. The impedance spectra of samples are represented in Fig. 6.

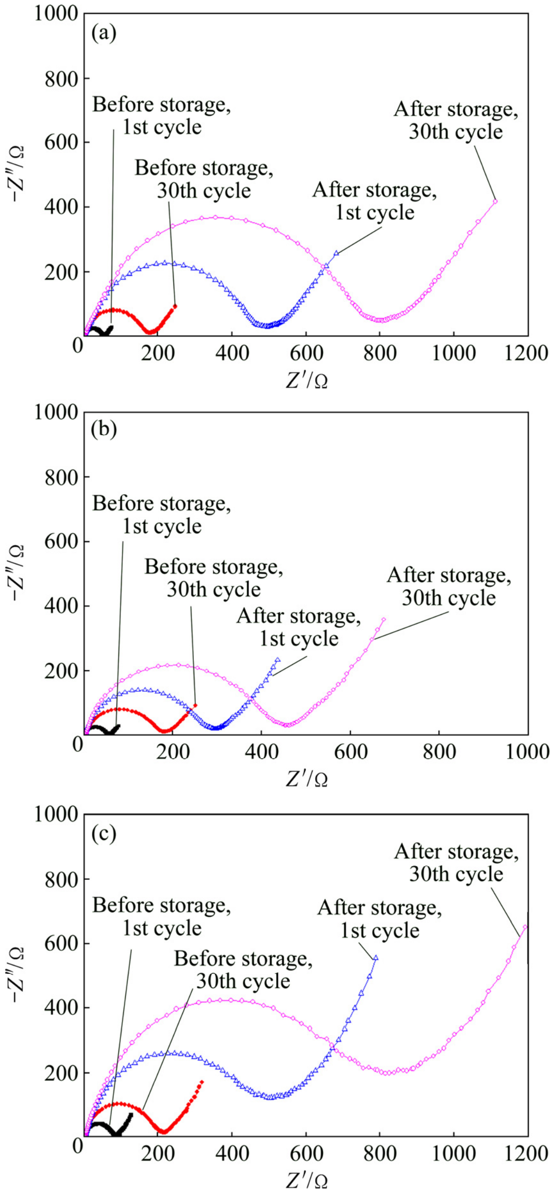

Fig. 6 EIS plots of samples F (a), EF2 (b) and ES2 (c) before and after storage in air for three months

Usually, an intercept in the high frequency region of the Z' real axis corresponds to the ohmic resistance (R_s), which represents the resistance of the electrolyte and electrode material. The semicircle in the middle frequency range indicates the charge transfer resistance (R_{ct}). The inclined line in the low frequency represents the Warburg impedance (Z_w), which is associated with lithium-ion diffusion in the material particles [19,20]. As shown in Fig. 6, compared with R_{ct} values, R_s values may be nearly neglected. Thus, R_{ct} values can be used to evaluate the electrochemical performance of the active material. Table 2 lists the R_{ct} values of samples after cycling for different times based on the EIS plots. For the sample F before storage, when the cycling times is increased from 1 to 30, its R_{ct} value increases from 55 to 171 Ω, equal to the increment rate of 3.87 Ω per cycle. However, after the sample F is stored in air with a relative humidity of 80% for three months, its R_{ct} value increases rapidly to 478 Ω after the 1st cycle. And the R_{ct} value of 30th cycle is 792 Ω, with the increment rate of 10.47 Ω per cycle. This indicates that the stored sample F suffers more resistance during the course of charge−discharge, which may be attributed to the formation of a large number of electrochemically inactive materials, including NiO-like species and absorbed hydroxyl and carbonate species (shown in Table 1) [7,15]. On the other hand, the fresh sample EF2 shows the similar R_{ct} values with the fresh sample F after charging/discharging for 1 cycle and 30 cycles. However, after the sample EF2 is stored for three months, its R_{ct} value increases only to 292 Ω after the 1st cycle. And the R_{ct} value increases by 159 Ω after the 30th cycle, with the increment rate of 5.3 Ω per cycle, nearly half of the corresponding value of the stored sample F. The smaller R_{ct} is in favor of rapid Li^+ diffusion and may result in better electrochemical performance of the active material (shown in Fig. 5). These data also suggest that the sample EF2 has better storage performance than the sample F, which has to ascribe to the removal of residual $LiOH/Li_2O$ with ethanol washing. As far as the sample ES2 is concerned, its R_{ct} values before and after storage display the similar increasing tendency to the sample F.

Table 2 R_{ct} values of samples F, EF2 and ES2 after different cycles

Sample	R_{ct}/Ω			
	Before storage		After storage	
	After 1 cycle	After 30 cycles	After 1 cycle	After 30 cycles
F	55	171	478	792
EF2	57	182	292	451
ES2	91	223	496	804

4 Conclusions

1) Ethanol washing can remove effectively the synthetic residues of $LiOH/Li_2O$ on the freshly-

synthesized $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ and make the powders much more resistant to H_2O and CO_2, without showing any bulk structural degradation and weakening the electrochemical performance.

2) The discharge specific capacity and cycle life of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ after storage in air with a relative humidity of 80% are improved by ethanol washing.

3) Ethanol washing without subsequent heat treatment is only used for the freshly-synthesized $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, which is different from water washing. And this method is also effective for other nickel-rich cathode materials.

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乙醇洗涤对 $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ 正极材料性能的影响

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摘　要： 采用乙醇溶剂对不同 $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ 正极材料进行洗涤。采用电感耦合等离子体原子发射光谱(ICP-AES)、傅里叶变换红外光谱(FTIR)、X 射线衍射(XRD)、扫描电子显微术(SEM)、充放电测试和电化学阻抗谱(EIS)对样品的元素含量、结构、形貌和电化学性能进行表征。结果表明，乙醇洗涤能够有效去除新制备的 $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ 表面残留的 $LiOH/Li_2O$ 杂质，不会破坏材料的本体结构、表面形貌和电化学性能，使得材料具有更好的耐 H_2O 和 CO_2 腐蚀性。并且，使用乙醇立即对新制备的材料进行洗涤，再置于相对湿度 80%的空气中储存 3 个月后，其放电比容量和循环性能相对于未洗涤样品得到大幅提升。

关键词： 锂离子电池；$LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$；乙醇洗涤；储存性能；电化学性能

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