A dataset in this report is regarding a research article “Crucial Role of Thioacetamide for ZrO_2 Coating on the Fragile Surface of Ni-rich Layered Cathode in Lithium Ion Batteries” [1]. Thioacetamide (TA) is introduced to form a homogeneous ZrO_2-coating in a facile method through washing with Zr(SO_4)_2 aqueous solution. The presence of the data in this paper indicated the role of TA for surface modification of LiNi_{0.82}Co_{0.09}Mn_{0.09}O_2 (NCM82) materials by ZrO_2, leading to improve the electrochemical performance of NCM82 Ni-rich cathode materials. These data were proceeded measurement electrochemical properties of cathode electrode on a battery cycler, the surface characteristics of the cathode materials were investigatated by SEM, EDS mapping, TEM and XPS. X-ray diffraction (XRD, Rigaku, SmartLab) was used to evaluate the influence of the coating layer on the microstructure of active materials.

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Specifications table

| Subject                      | Energy                                      |
|------------------------------|---------------------------------------------|
| Specific subject area        | Renewable Energy, Sustainability and the Environment |
| Type of data                 | Figure                                      |
| How data were acquired       | XRD, XPS, SEM, TEM, Lithium impurities, pH value, Voltage profiles, cycles and rate performance. |
| Data format                  | Raw and Analyzed                            |
| Parameters for data collection | pH value, the thickness of the ZrO₂ coating layer, applied current and voltage, cycle at room temperature and 45 °C and rate performance. |
| Description of data collection | Surface morphologies, pH condition, homogeneous coating, cycle and rate performance of the cells influenced by ZrO₂ coating layer on the cathode electrode. |
| Data source location         | Center for UI Research Facilities, Incheon National University, 12–1, Songdo-dong, Yeonsu-gu, Incheon, Republic of Korea. LAB, Department of Energy and Chemical Engineering, Incheon National University, 12–1, Songdo-dong, Yeonsu-gu, Incheon, Republic of Korea. Department of Chemistry, Research Institute of Basic, College of Science, Incheon National University, 12–1, Songdo-dong, Yeonsu-gu, Incheon, Republic of Korea. |
| Data accessibility           | The data are available with this article.   |
| Related research article     | Van-Chuong Ho, Seunghun Jeong, Taeeun Yim, Junyoung Mun, “Crucial Role of Thioacetamide for ZrO₂ Coating on the Fragile Surface of Ni-rich Layered Cathode in Lithium Ion Batteries”, J. Power Sources, https://doi.org/10.1016/j.jpowsour.2019.227625 |

Value of the Data

- This research is essentially understanding the role of TA in the surface modification for Ni-rich cathode materials by ZrO₂ via washing in Zr(SO₄)₂ aqueous solution.
- Data in this paper shows a homogeneous ZrO₂ layer formed on the surface of NCM82, leading to enhance the electrochemical performance of Ni-rich cathode materials.
- In this work, the TA has been controlling the pH value of the coating solution and supported Zr ions evenly distributed on the surface of Ni-rich cathode materials.
- This data provides a simple surface modification method for NCM Ni-rich cathode materials, low cost with ZrO₂ coated layer-based Zr-precursor and can be widely applied for coating technologies of transition metal oxide.

1. Data description

The data in this work has been derived by fabrication of the ZrO₂-coated layer by simple washing method. Figs. 1 and 2 show the structure change of host materials due to immersed in the coating solution. Fig. 3 shows the effect of different coating solution on the surface coating properties of the NCM82 cathode materials. Figs. 4 and 5 show the effect of coating solution on the surface morphologies of NCM82 materials, simultaneously description the thickness of ZrO₂ coating layer. Fig. 6 shows the amount of lithium impurities eliminated by coating solution and pH value of the coating solution was controlled by TA. Fig. 7 shows the effect of concentration of TA on the electrochemical performance of the ZrO₂-coated NCM82 Ni-rich cathode materials.

2. Experimental design, materials, and methods

The washing process and simultaneously surface coating by the aqueous solution is the simple method, the impurities on the surface of host materials are elimination and immediately Zr-precipitation to establish a solid protective layer for cathode materials. Many chemical and
physical parameters influence the overall process outcome. These include material parameter (pH value, Li delithiation, concentration, thickness and lattice parameter), electrochemical process (fading capacity, cycle and capacity). Experiment detail for the fabrication of ZrO$_2$-coated layer has been previously presented. The Zr(SO$_4$)$_2$•4H$_2$O, TA were dissolved in D.I water and NCM82 powders were added in the coating solution, stirred, filtered, dried filtered powders and calcination of black powder materials.

2.1. The effect of washing process on the structure change of NCM82 Ni-rich materials

The XRD data of all samples were performed measurement on the X-ray diffraction (XRD, Rigaku, SmartLab) with 2θ in a range from 10 to 80° and scanning mode of 0.3, (Cu target of 1.5412 Å). Fig. 1 shows the XRD spectra of the bare NCM82, NCM82 materials were washed in D.I water, dried at 80 ºC for 2 h (NCM-W80) and heated at 750 ºC for 3 h (NCM-W750), ZrO$_2$-coated NCM82 materials from Zr(SO$_4$)$_2$ solution without TA was dried at 80 ºC for 2 h (Z$_{0.08}$-80) and heated at 750 ºC for 3 h (Z$_{0.08}$-750). Fig. 1(a, b) show the XRD pattern of NCM-W80 and NCM-W750 compared with pristine NCM82 sample, observed that the peak at (003) of NCM-W80 shifts to lower 2θ (degree of shift peak, Δ(2θ) is 0.04°). Fig. 1(c, d) shows the XRD pattern of the Z$_{0.08}$-80 and Z$_{0.08}$-750, observed that peak at (003) of Z$_{0.08}$-80 had two 2θ peaks at 18.74° and 18.53°. The peak at (003) of the NCM-W750 and Z$_{0.08}$-750 shift to a higher angle close to the original peak position of the pristine sample, maybe due to the oxygen defects established in the octahedral structure of NiO$_6$ [2,3].
2.2. The effects of TA concentration of precursor chemical in coating solution on the structure change of the NCM82 Ni-rich materials

Fig. 2 shows the XRD properties dependence on the concentration of precursor chemical in the coating solution, Fig. 2(a, b) shown the XRD pattern of the NCM82 powders were immersed in the different concentration of Zr(SO$_4$)$_2$ of 0.04, 0.08, 0.16 and 0.24 M for 5 min, after filtered, dried and calcinated, the samples were named Z$_{0.04}$, Z$_{0.08}$, Z$_{0.16}$ and Z$_{0.24}$, respectively. The peak at (003) is moved to the higher angle $2\theta$ when increases concentration of Zr in the coating solution, due to the amount of Zr-coated increased. The Fig. 2(c, d) show the XRD spectra of coating sample from 0.08 M Zr(SO$_4$)$_2$ with different TA concentration of 0, 0.1, 0.2, 0.3 and 0.4 M compared pristine sample, the samples were named Z$_{0.08}$, Z$_{0.08}T_{0.01}$, Z$_{0.08}T_{0.2}$, Z$_{0.08}T_{0.3}$ and Z$_{0.08}T_{0.4}$, respectively. The peak at (003) is shifted to the higher angle $2\theta$ when increases concentration of TA in the coating solution, TA could be agent controlling thickness of ZrO$_2$-layers as well as the amount of Zr-precipitation on the host materials.

2.3. The characterization of surface modification for Ni-rich layered oxide cathode materials

The chemical composition on the surface of the pristine NCM82, Z$_{0.08}$ and Z$_{0.08}T_{0.2}$ were determined by XPS analysis as shown in Fig. 3, all XPS spectra were calibrated against the C 1 s
Fig. 3. The XPS patterns of the ZrO$_2$-coated NCM82 Ni-rich material was calcinated at 750 °C for 3 h in oxygen. In this experiment, 0.08 M Zr(SO$_4$)$_2$ with 0.2 M TA and without TA was used as a coating solution. (a) after Ar-etching 3 s, (b) after Ar-etching 30 s and (c) XPS peaks of Ni 2p, Co 2p, Mn 2p and S 2p of bare, Z$_{0.08}$ and Z$_{0.08}T_{0.2}$ without Ar-etching, (d) the fitting Ni 2p, Co 2p, Mn 2p XPS spectra of bare, Z$_{0.08}$ and Z$_{0.08}T_{0.2}$ without Ar-etching.

The XPS measurement has proceeded after Ar-etching 3 s (Fig. 3(a)) or 30 s (Fig. 3(b)). Z$_{0.08}$ and Z$_{0.08}T_{0.2}$ show the characterization peaks at 184.10 eV (Zr 3d$_{3/2}$) and 181.68 (Zr 3d$_{5/2}$), which was observed in the Z 3d spectra, and intensity of the Zr 3d$_{3/2}$ and Zr 3d$_{5/2}$ peaks with Z$_{0.08}T_{0.2}$ is much higher than that these peaks in Z$_{0.08}$ sample at the same condition [1]. The one of the characterization of lithium impurity (Li$_2$CO$_3$) was observed in O 1s spectrum (at 531.8 eV), the intensity of this peak in is decreasing for Z$_{0.08}$ and Z$_{0.08}T_{0.2}$ compared with the bare sample.
2.4. Surface morphologies analysis of bare sample, Z0.08 and Z0.08T0.2

Surface morphologies of the bare NCM82, Z0.08 and Z0.08T0.2 were shown in Fig. 4(a), the ZrO2 coating layer on the surface of Z0.08T0.2 is relatively uniformity, meanwhile, a coating materials layer on the surface of Z0.08 is un-even distributed. For the bare sample, the white grain was observed that is the lithium impurities as a report of X. Xiong et al. [3]. In addition, Zr element is more evenly distributed on the surface of the Z0.08T0.2 sample compared with bare sample [1].

2.5. Effect of coating solution on the distribution ZrO2-thick coating layer

Fig. 5(a) shows the distribution thickness of the ZrO2 on the surface of the Z0.08, Z0.08T0.2 and Z0.08T0.3, which was collected from TEM image Fig. 5(b, c, d). The thickness of coating materials layer was calculated by two software of excel (follow Pythagoras's theorem as described in Fig. 5(c)) and directly from ImageJ software. The ZrO2-thick is relative uniformity, which varied from 5.06 to 7.08 nm and the average thickness is about 5.75 nm. A ZrO2 layer on the surface of Z0.08 is varied from 1.42 to 3.96 nm and an average thickness of 2.75 nm. For the Z0.08T0.3 sample, the thickness of a ZrO2 layer varied from 5.9 to 13.4 nm and an average thickness of about 9.68 nm.

2.6. Effect of pH value of coating solution on the solubility of the lithium impurities on the surface of Ni-rich layered cathode structure

Fig. 6(a) shows the amount of Li impurities (LiOH/ Li2CO3) with bare NCM82, washed NCM82, Z0.08 and Z0.08T0.2 sample. The concentration of Li impurities on the surface of the sample is decreased when the amount of TA increases in the coating solution. Videlicet, TA has supported ability dissolution of Li impurities and controlled pH value of coating solution as shown in Fig. 6(b). Concretely, amount of Li impurities were eliminated in a washed sample of (29.02% Li2CO3, 80.74% LiOH), in Z0.08 is (33.7% Li2CO3, 82.96% LiOH) and in Z0.08T0.2 is (80.82% Li2CO3, 85.15% LiOH), compared with the bare sample.
Fig. 5. The thickness of the ZrO$_2$ coating layer was carefully determined by ImageJ software and simple excel method. (a) The thickness distribution forms on the surface of the Z$_{0.08}$ and Z$_{0.08}$T$_{0.2}$ sample, (b) TEM image of Z$_{0.08}$, (c) TEM image of Z$_{0.08}$T$_{0.2}$ and (d) TEM image of Z$_{0.08}$T$_{0.3}$.

Fig. 6. (a) Lithium impurities were performed measurement by titration method on Titrino Plus 848 machine, (b) pH value of coating solution as a function of TA concentration.
Fig. 7. Effect of concentration of TA on the electrochemical performance of the ZrO$_2$-coated NCM82 Ni-rich cathode materials. (a) initial charge/ discharge voltage profiles at 25 °C, (b) specific discharge capacity at 25 °C, (c) specific discharge capacity of bare NCM82 and $Z_{0.08}T_{0.2}$ sample at 45 °C and (d) rate performance of the bare NCM82 and $Z_{0.08}T_{0.2}$ at 25 °C. The half-cells were cycled at 0.3 C after formation cycles 0.2 C and a voltage of 2.5–4.25, (1 C = 200 mA g$^{-1}$).

2.7. Electrochemical performance

Electrochemical properties of the ZrO$_2$-coated NCM82 by different concentration of Zr(SO$_4$)$_2$ without TA has been reported in a previous paper [Ref]. In this report, the electrochemical performance of $Z_{0.08}T_x$ (0 ≤ x ≤ 0.4) and bare NCM82 were showed in Fig. 7, the half-cells were cycled at 0.3 C after formation cycles 0.2 C and a voltage of 2.5 – 4.25, (1 C = 200 mA g$^{-1}$). Fig. 7(a) compares the charge/ discharge voltage profiles of the $Z_{0.08}$, $Z_{0.08}T_{0.1}$, $Z_{0.08}T_{0.2}$, $Z_{0.08}T_{0.3}$ and $Z_{0.08}T_{0.4}$ samples at 0.2 C and a cut-off voltage of 2.5–4.25 V. Although $Z_{0.08}T_{0.1}$ shows the highest specific capacity of 196.8 mAh g$^{-1}$ for the initial cycle, it shows poor cycle ability. The $Z_{0.08}T_{0.2}$ sample shows the highest capacity retention of 89.35% after 100 cycles and 76.69% after 190 cycles. The $Z_{0.08}T_{0.2}$ sample is also shown good cyclability at 45 °C and higher rate performance compared with bare NCM82 (Fig. 7(c, d)).

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.dib.2020.105458.

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