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Aluminum and zirconium dual-doped nickel-cobalt-zinc oxide cathode material for lithium-ion batteries

Lityum iyon piller için alüminyum ve zirkonyum çift katkılı nikel-kobalt-çinko oksit katot malzemesi

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Aluminum and Zirconium Dual-Doped Nickel-Cobalt-Zinc Oxide Cathode Material for Lithium-Ion Batteries

Highlights

- *The synthesis of cathode materials with a sol-gel-assisted solid-state doping method.*
- *Production of dual-doped cathode materials.*
- *Obtaining the layered oxide structure.*
- *Successful incorporation of aluminum and zirconium into the layered oxide structure.*
- *Structural analysis.*

Graphical Abstract

Aluminum (Al) and zirconium (Zr) were incorporated into nickel-cobalt-zinc ternary hydroxide material ($NiCoZn(OH)_2$) to produce dual-doped $Li_{1.05}Ni_{0.8}[Co_{0.054}Zn_{0.046}]Al_{0.08}Zr_{0.02}O_2$ (NiCoZn –AlZr) cathode material.

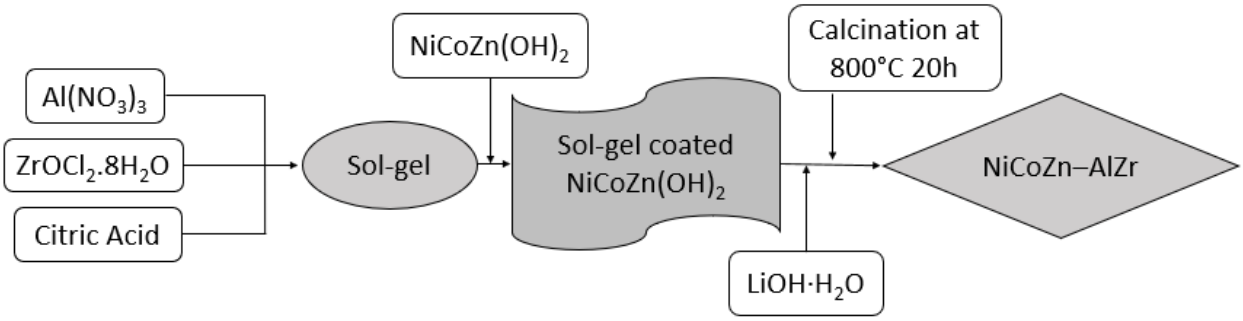

Figure. Schematic representation of the sol-gel assisted solid-state doping method.

Aim

The study aims to synthesize and characterize doped cathode material by successfully incorporating Al and Zr into the structure.

Design & Methodology

Al and Zr-doped cathode materials were synthesized using the sol-gel-assisted solid-state doping method, and characterization was done using XRD, Rietveld refinement, SEM-EDS, ICP-MS, XRF, and CV analysis.

Originality

Unlike the literature, our study incorporated aluminum and zirconium into a nickel-cobalt-zinc ternary hydroxide using the sol-gel-assisted solid-state doping method.

Findings

A 20-hour calcination process at 800 degrees Celsius resulted in a peak intensity ratio of $I_{(003)}/I_{(104)} = 1.2$, and the c/a value was found to be 4.955. These structural parameters are critical to the material's electrochemical stability and charge/discharge cycling performance.

Conclusion

The active material synthesized in this study demonstrates promising characteristics for advanced lithium-ion battery applications. A systematic investigation of Al and Zr doping effects on the NiCoZn –AlZr structure may provide valuable insights into designing and optimizing next-generation cathode materials with improved stability and performance.

Declaration of Ethical Standards

This article's author(s) declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Aluminum and Zirconium Dual-Doped Nickel-Cobalt-Zinc Oxide Cathode Material for Lithium-Ion Batteries

(Bu çalışma ICMATSE 2024 konferasında sunulmuştur. / This study was presented at ICMATSE 2024.)

Araştırma Makalesi / Research Article

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ABSTRACT

Aluminum (Al) and zirconium (Zr) doping have been found to enhance the structural and electrochemical performance of cathode materials, potentially improving their suitability for lithium-ion battery applications. Therefore, in this study, Al and Zr were incorporated into nickel-cobalt-zinc hydroxide material via sol-gel assisted solid-state doping synthesis to produce dual-doped $Li_{1.05}Ni0.8[Co_{0.054}Zn_{0.046}]$ $Al_{0.08}Zr_{0.02}O_2$ (NiCoZn–AlZr) cathode material. The synthesized powders were characterized using SEM-EDS, XRD, and Rietveld refinement analysis. As a result of the calcination process lasting 20 hours at 800 degrees, the XRD peak intensity ratio $I_{(003)}/I_{(104)}$ was 1.2, and the c/a value was 4.955. These structural parameters are critical to the material's electrochemical stability and charge/discharge cycling performance. Additionally, the cyclic voltammetry test proved the redox reversibility of the cathode material. In conclusion, this study's NiCoZn–AlZr cathode material demonstrates promising characteristics for advanced lithium-ion battery applications. A systematic survey of the doping process for NiCoZn–AlZr can provide valuable information for designing next-generation cathode materials with improved stability and performance.
Keywords: Lithium-ion battery, cathode material, energy storage.

Lityum İyon Piller için Alüminyum ve Zirkonyum Çift Katkılı Nikel-Kobalt-Çinko Oksit Katot Malzemesi

ÖZ

Alüminyum (Al) ve zirkonyum (Zr) katkılamasının, katot malzemelerinin yapısal ve elektrokimyasal performansını artırdığı, potansiyel olarak lityum iyon pil uygulamaları için uygunluklarını geliştirdiği tespit edilmiştir. Bu nedenle, bu çalışmada Al ve Zr, sol-jel destekli katı hal katkılama sentezi yoluyla nikel-kobalt-çinko hidroksit malzemeye birlikte katkılanarak çift katkılı $Li_{1.05}Ni_{0.8}[Co_{0.054}Zn_{0.046}]$ $Al_{0.08}Zr_{0.02}O_2$ (NiCoZn–AlZr) katot malzemesi üretilmiştir. Sentezlenen tozlar SEM-EDS, XRD ve Rietveld analizleri ile karakterize edilmiştir. 800 derecede 20 saat süren kalsinasyon işlemi sonucunda $I_{(003)}/I_{(104)}$ XRD pik şiddetleri oranı 1,2 ve c/a değeri 4,955 olarak bulunmuştur. Bu yapısal parametreler, malzemenin elektrokimyasal kararlılığı ve şarj/deşarj döngüsü performansının kritik göstergeleridir. Ayrıca çevrimsel voltametri testi ile katot malzemesinde gerçekleşen redoks reaksiyonlarının tersinebilir olduğu kanıtlanmıştır. Sonuç olarak, sentezlenen NiCoZn–AlZr katot malzemesi, gelişmiş lityum iyon pil uygulamaları için ümit verici özellikler göstermektedir. NiCoZn–AlZr'nin katkılama işleminin sistematik bir şekilde incelenmesi, gelişmiş kararlılık ve performansa sahip yeni nesil katot malzemelerinin tasarlanmasında değerli bilgiler sağlayabilir.
Anahtar kelimeler: Lityum iyon pil, katot malzemesi, enerji depolama.

1. INTRODUCTION

Rechargeable (secondary) lithium-ion batteries (LIBs) are frequently used in energy storage systems due to their high energy density, fast response, and high cycle efficiency [1,2]. The most important factors limiting the ability of LIBs to reach higher energy and power densities are undesirable solid-electrolyte reactions, crack formation, and degradation. Most current cathode materials are inadequate to meet the rising energy demands due to their low energy density associated with these problems [3,4]. Many studies utilizing doping and/or surface modification techniques are being applied to prevent these issues and increase the active materials' performance. The doping process can alleviate the structural instability of the electrode by restricting the movement of transition metals and preventing oxygen loss. Al^{3+}, Mg^{2+}, Zr^{4+}, Cr^{3+}, and Ti^{4+} are commonly used ions for doping [5–8].

Research shows that doping Al, a transition element, reduces cation mixing and improves the structural and thermal stability of batteries [9]. On the other hand, Zr doping enhances cycling stability and specific capacity [10]. Moreover, co-doping with Al and Zr minimizes cation mixing and microcrack formation [11,12] by creating synergistic effects. Among various additive candidates, Zn is recommended as a promising element

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for new studies due to its affordable cost and the similarity of the K_{sp} value (solubility product constant) of $Zn(OH)_2$ (3.0×10^{-17}) to that of $Ni(OH)_2$ (5.5×10^{-15}). In addition, the ionic sizes of Li^+ and Zn^{2+} are very close (r_{Li^+} = 76 pm and $r_{Zn^{2+}}$ = 74 pm), indicating that Zn ions are likely to be found in Li regions.

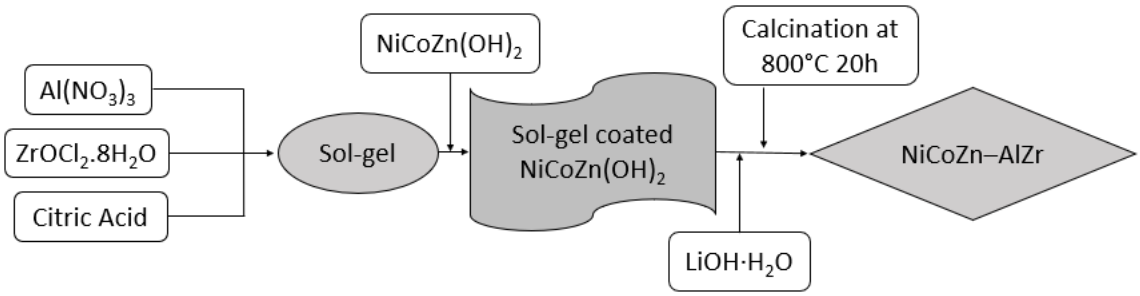

Figure 1. Schematic representation of the sol-gel assisted solid-state doping method

Therefore, it is suggested that, similar to Mg^{2+}, which is known to be present on Li sites, Zn^{2+} can act as a pillar ion to prevent and/or delay lattice collapse [6]. Cui et al. [6] synthesized $LiNi_{0.94}Co_{0.04}Zn_{0.02}O_2$ (NiCoZn) cathode material by doping Zn into high Ni content $LiNi_{0.94}Co_{0.06}O_2$. As a result of the study, the discharge capacity and cycle life of the batteries

were increased. However, the impact of multi-cation doping on the structure and performance of NiCoZn cathode active materials requires more attention.

Many methods are used for producing cathode powder, such as co-precipitation, sol-gel, spray drying, solid-state synthesis, and microwave synthesis [13–15]. A relatively new method referred to as the sol-gel supported solid-state doping method reported by Kong et al. [12], Cao et al. [16], and Shen et al. [17], gave successful results in the synthesis of Al-doped $LiNiO_2$, Al/Mg-doped $LiNiO_2$, and Al/Zr-doped $LiNiO_2$ materials, respectively. In this specific solid-state doping method, the surface of the spherical nickel-based hydroxide starting powders is modified with a sol-gel containing the doping elements by forming a uniform and continuous coating layer, and the doping elements are incorporated homogeneously into the structure by interdiffusion as a result of the calcination process [16,18]. The secondary grain sizes of the powders obtained with this method are also in the micrometer range, and the powders show good sphericity [12,16,17,19].

In this study, we have synthesized $Li_{1.05}Ni_{0.8}[Co_{0.06}Zn_{0.04}]Al_{0.08}Zr_{0.02}O_2$ (NiCoZn–AlZr) cathode material using a sol-gel assisted solid-state doping method (Figure 1). Following calcination at 800 °C for 20 hours, the materials underwent analysis via SEM-EDS, XRF, ICP-MS, XRD, and Rietveld refinement.

In addition, the reversibility of the cathode material was examined by a cyclic voltammetry test.

2. MATERIALS and METHOD

Appropriate amounts of zirconium (IV) oxychloride octahydrate ($ZrOCl_2.8H_2O$, Acros Organics) and aluminum nitrate nanohydrate ($Al(NO_3)_3.9H_2O$, Merck) were dissolved in distilled water. Then, citric acid, a gelling agent, was added to the solution. Spherical nickel (II) based hydroxide ($NiCoZn(OH)_2$, Nanografi) was added to the obtained sol-gel, and the mixing process was continued for 24h. After this process, it was subjected to a rapid drying process at 150 °C. The obtained powder was mixed with lithium hydroxide ($Li(OH)_2 \cdot 2H_2O$) in an agate mortar until it became homogeneous [17]. To get an Al/Zr-modified cathode material, NiCoZn–AlZr, the calcination process was performed at 800 °C for 20 h. The accuracy of the synthesized materials formula was assessed using an Olympus Vanta M Series X-ray fluorescence (XRF) analyzer and a Thermo Scientific iCAP RQ inductively coupled plasma–mass spectrometer (ICP-MS). X-ray diffraction (XRD) analysis with Bragg angles (2θ) between 10–70° was performed using a PANalytical X-Pert3 Powder (Cu Kα beam source) diffractometer. Lattice parameters (*a*, *c*) and oxygen position (*Z*) were determined by the Rietveld analysis (Match! 4 software). Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were analyzed using a Zeiss EVO LS 10 instrument. The cathode active material was mixed with carbon black and PVDF (dissolved in NMP) (mass ratio = 8:1:1) to make a homogeneous slurry and spread on aluminum foil. After this process, the electrode was dried at 100 °C for 12 hours in a vacuum oven. Lithium anode and glass fiber separator were used to fabricate CR2032-type coin cells in a glove box. 1 mol $LiPF_6$ dissolved in a 1:1:1 solution of EC:DMC:EMC was used as the electrolyte solution. Cyclic voltammetry analysis (CV) curves of the installed batteries were obtained using a battery test system (BaSyTec) in a 2.7–4.3 V voltage window at 25 °C.

3. RESULTS AND DISCUSSIONS

Table 1 shows XRF and ICP-MS measurements of the synthesized material. The measurements are in good agreement, and the elements' mole ratios were close to their theoretical values: $Li_{1.05}Ni_{0.8}[Co_{0.054}Zn_{0.046}]Al_{0.08}Zr_{0.02}O_2$. Slight differences in the chemical compositions determined by the two methods may arise from the instrumental detection limits

and issues related to sampling and analysis, such as sample contamination during collection, processing, transporting, weighing, and analyzing procedures [20]. Since the elemental analysis by XRF is closer to the theoretical calculations, the XRF measurements are taken as the basis for the rest of the study, in which the chemical formula of the material was determined as $Li_{1.05}Ni_{0.8}[Co_{0.054}Zn_{0.046}]Al_{0.08}Zr_{0.02}O_2$.

Figure 2a shows the XRD spectrum of the synthesized NiCoZn–AlZr cathode material exhibits a similar diffraction pattern with the α-$NaFeO_2$ type hexagonal structure belonging to the R-3m space group (ICDD card no. 96-152-0790) [16,21]. The separation of the $I_{(006)}/I_{(102)}$ and $I_{(108)}/I_{(110)}$ peaks marked in the spectrum represents a well-ordered layered structure [22]. This indicates that the doping elements were correctly incorporated without disrupting the structure and that easy entry/exit of lithium ions is allowed during charging/discharging [10,23]. No impurity peaks were detected in the XRD spectrum, which also confirms that both Al and Zr can be doped into Li(NiCoZn)O_2-based layered structure [11,24–26]. During the material synthesis, cation mixing occurs if Ni^{2+} ions occupy the Li^+(3b) sites and Li^+ ions occupy the Ni^{2+}(3a) sites in the layered crystal lattice.

Table 1. ICP-MS and XRF results of the synthesized material

Analysis	Ni	Co	Zn	Al	Zr	Chemical Formula
XRF (ppb)	508.42	34.64	33.99	22.968	16.501	$Li_{1.05}Ni_{0.8}[Co_{0.054}Zn_{0.046}]Al_{0.08}Zr_{0.02}O_2$
% mol	80.2%	5.4%	4.6%	7.9%	1.7%	
ICP-MS (ppb)	559.14	40.80	35.98	25.83	10.833	$Li_{1.05}Ni_{0.8}[Co_{0.058}Zn_{0.046}]Al_{0.08}Zr_{0.02}O_2$
% mol	79.7%	5.80%	4.61%	8.01%	1.84%	

The higher this ratio, the lower the degree of Li^+/Ni^{+2} cation mixing [27,28]. The intensity ratio of the (003) and (104) peaks, ($I_{(003)}/I_{(104)}$), is an essential criterion in determining the degree of cation mixing, which has detrimental effects on electrochemical lithium intercalation reactions. The minimum acceptable value was reported as 1.2 [16,17,23,30]. This value was also found to be 1.2 in this study. In addition, the ratio of the sum of the intensity values of the peaks (006) and (102) to the intensity of the peak (101), ($R=(I_{(006)}+I_{(102)})/I_{(101)}$), is an indicator of good hexagonal ordering. An R-value below 0.5 indicates a well-layered hexagonal structure [23,27,31]. In the obtained XRD graphs, the R-value was calculated as 0.48. These results confirm the successful incorporation of aluminum and zirconium and demonstrate the effectiveness of the adopted doping approach.

Crystallographic data (c/a, lithium, and metal-oxide gaps) for the NiCoZn–AlZr cathode material were obtained from the Rietveld refinement analysis presented in Figure 2b and Table 2. Tan et al. [19] stated that the c/a ratio, reflecting the degree of cation order, must be greater than 4.899. Table 2 shows that this value was 4.955 in the current study. Additionally, lithium layer spacing ($D(LiO_2)$) and metal layer spacing ($D(MO_2)$) values are pretty crucial in terms of facilitating Li^+ de/intercalation during charge/discharge cycles [10,30]. It was determined that $D(LiO_2)$ is wider than $D(MO_2)$ (Table 2).

Table 2. Results of the Rietveld refinement analysis (**c/a**: degree of cation order, **$\mathcal{Z}$**: oxygen position, **$D(MO_2)$**: metal layer spacing, and **$D(LiO_2)$**: lithium layer spacing)

Sample	NiCoZn–AlZr
c/a	4.955
$\mathcal{Z}$	0.25867
$D(MO_2)$[a] (Å)	2.1247
$D(LiO_2)$[b] (Å)	2.6182

[a] $D(MO_2)= 2c\ (1/3 - \mathcal{Z})$

[b] $D(LiO_2)= c/3 - (D(MO_2)$

This is attributed especially to the addition of Zr because the bond energy of Zr-O (760 kJ mol^{-1}) is much higher than other transition elements, leading to an enlarged Li layer spacing [10]. $D(MO_2)$ and $D(LiO_2)$ values were calculated as 2.1247 Å and 2.6182 Å, respectively, which are consistent with the literature [26].

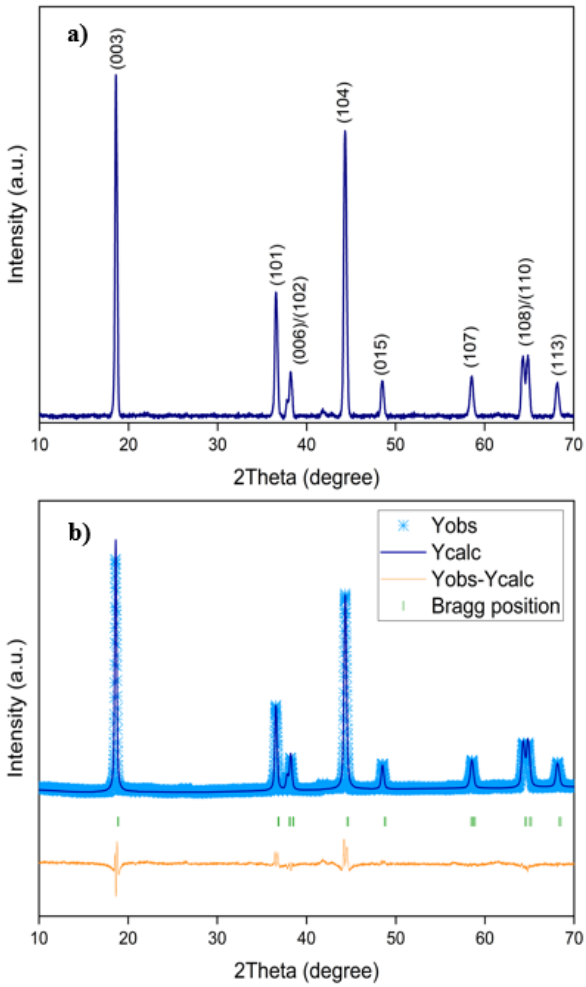

Figure 2. a) XRD spectrum, and b) Rietveld refinement analysis of the synthesized NiCoZn-AlZr cathode material. The experimental pattern is presented by the blue data points (Y_{obs}), the calculated pattern is given by the solid blue line (Y_{calc}), and the difference between the observed and calculated patterns is presented by the solid orange line (Y_{obs}-Y_{calc}). The vertical green ticks indicate the Bragg positions for an α-$NaFeO_2$-type hexagonal structure [29]

Figure 3 shows SEM images of the starting hydroxide and the synthesized NiCoZn –AlZr materials. These images clearly show that the spherical form is not distorted during calcination and doping processes. Figure 3b shows that the NiCoZn–AlZr cathode material consists of secondary particles surrounded by primary crystals at the nanometer scale. Figure 4 shows the cross-sectional linear element analysis of NiCoZn–AlZr powders. When the obtained spectrum is examined, it is seen that Ni is present in the structure in the highest amount (80%), and Al and Zr are included in the structure homogeneously throughout the particle. The cyclic voltammogram presented in Figure 5 provides insight into the electrochemical behavior and reversibility of the charge/discharge reactions of the cathode material.

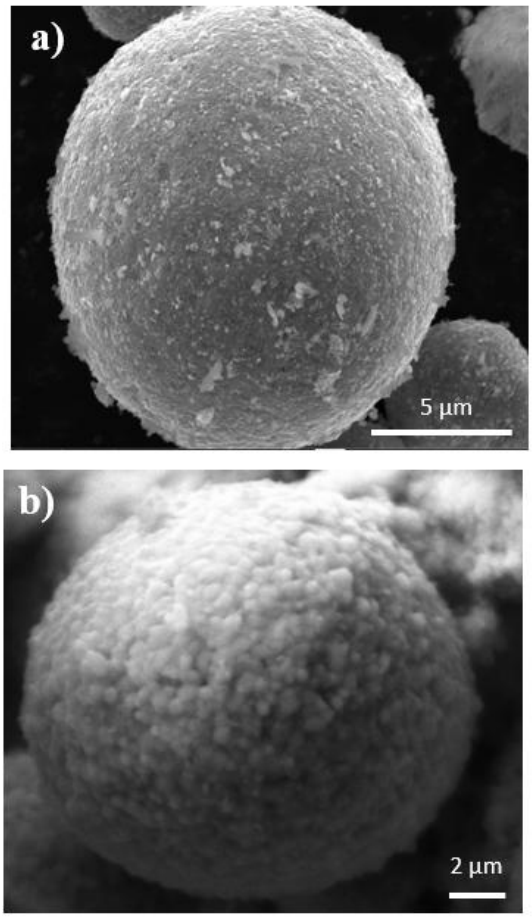

Figure 3. SEM images of a) spherical $NiCoZn(OH)_2$ starting powder and b) synthesized NiCoZn–AlZr powder

$LiNiO_2$ structures generally have three redox couples in the range of 2.7-4.3V. Each redox peak indicates the phase transformation that occurs due to the movement of Li^+ during charge-discharge. Phase transformations during charging occur as hexagonal to monoclinic ($H_1 \rightarrow M$) ($Ni^{3+} \rightarrow Ni^{4+}$) at ~3.7V, monoclinic to second hexagonal ($M \rightarrow H_2$) at 4V, and second hexagonal to third hexagonal ($H_2 \rightarrow H_3$) at 4.2V [16,32–34]. The oxidation peak at 3.95 V in the first cycle shifted to the left and decreased to 3.7 V from the second cycle onwards (Table 3), which can be attributed to the formation of the solid-electrolyte interface [11].

ΔE (0.08V) shown in Figure 5 indicates the amount of shift between oxidation (Epa) and reduction (Epc) peaks, and generally, the smaller this value is, the better the reversibility of charge discharge [10].

Umair et al. [35] found this value as 0.119V in the $Li_{1.1}Co_{0.8}Al_{0.075}Zr_{0.025}O_2$ cathode material obtained by doping 0.75 mol Al and 0.025 mol Zr. In their study, Ni et al. [10] found the oxidation potential as 3.72V and the reduction potential as 3.65V for the $LiNi_{0.9}Al_{0.09}Zr_{0.01}O_2$ cathode material. They calculated the ΔE value as 0.07V [10]. Therefore, it can be said that the obtained result is compatible with the findings of the literature.

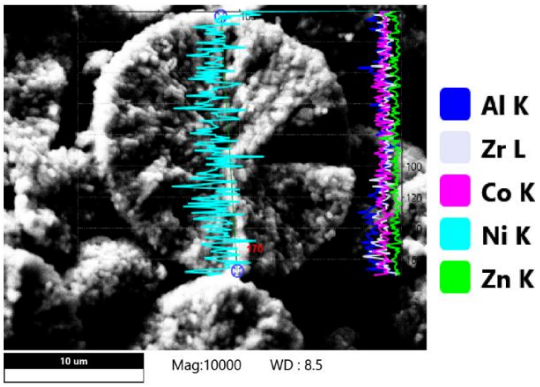

Figure 4. Cross-section EDS linear scan profile of NiCoZn-AlZr

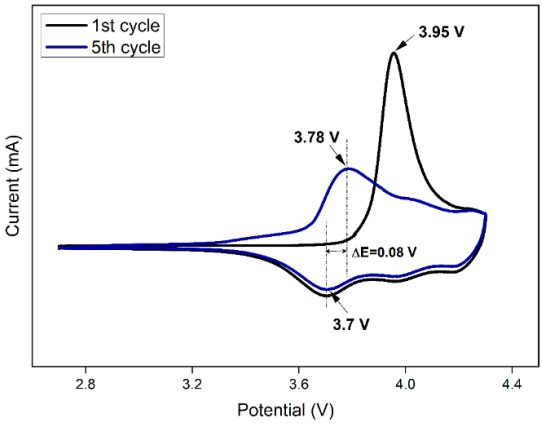

Figure 5. CV curves of NiCoZn–AlZr

Table 3. CV peak positions of NiCoZn–AlZr

	Cycle number	Epa (V)	Epc (V)
NiCoZn-AlZr	1st cycle	3.95	3.7
	5th cycle	3.78	3.7

4.CONCLUSION

The Al and Zr co-doped NiCoZn cathode material synthesized via the sol-gel-assisted solid-state doping method exhibits promising structural and electrochemical advantages for next-generation lithium-ion battery systems. XRD and Rietveld refinement analyses confirmed that the dual-doping strategy preserves a highly ordered layered framework with minimal cation mixing after calcination at 800 °C for 20 hours. The lithium layer spacing obtained in this study aligns well with the benchmark values reported in the literature, supporting efficient Li^+ de/intercalation during charge–discharge processes. Furthermore, cyclic voltammetry analysis underscored the capability of the NiCoZn–AlZr structure to maintain stable electrochemical kinetics. The combined effects of Al^{3+} and Zr^{4+} introduce a clear synergistic enhancement: Al contributes to improved lattice stability by suppressing cation disorder, while Zr promotes Li-layer expansion due to its strong Zr–O bond strength, collectively strengthening both structural integrity and ion transport pathways. These findings establish the NiCoZn–AlZr composition as a particularly promising candidate within the family of modified Ni-based layered oxides.

Overall, the results demonstrate that Al and Zr co-doping provides an effective pathway to engineer higher stability, improved reversibility, and enhanced functional performance in NiCoZn-based cathode materials. A systematic exploration of this dual-doping strategy has strong potential to guide the development of advanced cathodes with superior electrochemical durability. Future work will include comparative capacity retention and rate capability studies to further reveal and optimize the performance potential of the NiCoZn–AlZr material.

ACKNOWLEDGEMENT

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DECLARATION OF ETHICAL STANDARDS

This article's author(s) declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Duygu CANDEMIR planned the experiments, carried out the synthesis, processed the experimental data, conducted the analysis, wrote the main text, and edited the figures.

Oncu AKYILDIZ participated in the planning and supervision of the study, helped with the interpretation of the results, and made final checks on the manuscript.

Hakan ATES assisted with planning, supervised the study process, and collaborated on the manuscript by helping to interpret the results. All authors discussed the results together, reflected on the manuscript, and contributed to the final version of the paper.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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