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Overview of oxide electrode materials for lithium–ion batteries

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Abstract. The article discusses the prospects for developing lithium-ion batteries, emphasizing lithium-enriched transition metal oxides used as cathode materials for lithium-ion batteries (LIB). The primary focus is on materials with the formula $xLi_2MnO_3{\cdot}(1-x)LiMO_2$ (where M=Mn, Ni, and Co) that exhibit high discharge capacity (over 250 mAh/g) and specific energy (over 950 Wh/kg), surpassing traditional cathode materials such as $LiCoO_2$, $LiMn_2O_4$, and $LiFePO_4$. These oxides combine the monoclinic phase of Li_2MnO_3 and the trigonal phase of $LiMO_2$, which ensures their high performance. However, the authors note several problems, including low speed characteristics, irreversible capacity of the first cycle, and degradation of voltage and capacity during cycling. These problems are linked to the creation of spinel-like structures, unwanted reactions at the surface with the electrolyte, and the release of oxygen. The authors propose modification methods like protective coatings, alloying, and the creation of composite structures to enhance the characteristics. The article also includes an overview of other common cathode materials such as $LiCoO_2$, $LiMn_2O_4$, $LiNiO_2$ and their combinations, highlighting their advantages and limitations. Special attention is paid to promising materials, including $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ and $LiFePO_4$, which have balanced electrochemical and economic properties. It was also emphasised that further research is needed to understand the degradation mechanisms and optimise the structure of lithium-enriched oxides. Resolving these issues can help create better and more reliable cathode materials for LIB, which is crucial for advancing electric vehicles and other energy-intensive technologies.

Keywords: *spent automotive catalysts, rare metals, precious metals, rare earth elements, platinum metals, hydrometallurgical technologies.*

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1. Introduction

The history of lithium-ion batteries (LIB) originates in the discoveries of Alessandro Volta and Luigi Galvani in electrochemistry in the early 19th century, which initiated the research of chemical current sources. A significant contribution was made by Michael Faraday, who developed the laws of electrolysis and introduced the term “ion”.

Early rechargeable batteries such as lead-acid, nickel-cadmium, and nickel-iron served as the basis for further development. LIB's commercial breakthrough came in 1990, when Sony introduced batteries with graphite anodes and cathodes from $LiCoO_2$, contributing to the development of electric mobility. Despite the achievements, LIB research continues to improve the materials for the cathode, anode, electrolyte, and other components to increase productivity and reduce cost, as evidenced by the large number of publications and conferences on this topic.

Special attention is paid to cathode materials, where it is necessary to increase the specific capacitance, expand the cycling voltages and ensure stability during long-term cycling. It is essential to find cheaper and environmentally friendly alternatives to the toxic and expensive $LiCoO_2$, which is widely used in industry. $LiMn_2O_4$ spinel is considered a promising cathode material due to its low cost, low toxicity and high thermal stability, especially in the charged state. However, the known disadvantages of $LiMn_2O_4$, such as unsatisfactory cyclability and poor reproducibility, require further research and optimisation, including cationic and anionic doping, changes in stoichiometry, and surface modification.

2. Materials and methods

2.1. Overview of widely used oxide cathode materials of lithium–ion batteries

During the development of cathode materials for lithium-ion batteries (LIB), lithium cobaltate $LiCoO_2$ turned out to be the most effective, which is still widely used in the industrial production of LIB [1]. This compound has a layered structure similar to α-$NaFeO_2$ or the structure of rock salt. The crystal structure of $LiCoO_2$ consists of densely packed layers of cobalt (Co^{3+}) and lithium (Li^{+}) ions alternating along the (111) plane. Since such an ordering (111) violates the hexagonal symmetry of the lattice, $LiCoO_2$ crystallises in the space group R3m with unit cell parameters $a = 2.816$ Å and $c = 14.08$ Å. Figure 1 shows a schematic representation of the crystal structure of $LiCoO_2$, illustrating the layered arrangement of lithium, cobalt, and oxygen atoms.

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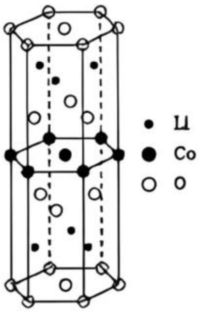
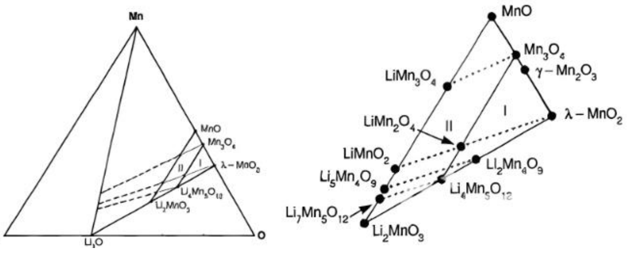


Figure 1. Schematic representation of the $LiCoO_2$ structure [2]

The theoretical capacity of lithium cobaltate ($LiCoO_2$) during electrochemical cycling is 274 mAh/g in the voltage range of 2.5-4.3 V. However, a significant disadvantage of this material is that its practical capacity is theoretically limited since the structure of $Li1–xCoO_2$ is not stable when lithium is deintercalated to $x > 0.5$. This limitation and the high cost of cobalt served as an incentive to search for alternative cathode materials for lithium-ion batteries with higher electrochemical capacity, energy and better resistance to cycling [2]. Lithium-manganese spinel $LiMn_2O_4$ has become one of the successful materials developed. The Li-Mn-O system is more complex than the Co-O system and includes more than ten stable compounds with different structures and degrees of manganese oxidation. Figure 2 shows the isothermal cross-section of the Li–Mn–O phase diagram and an enlarged fragment indicating stoichiometric compositions with rock salt, spinel, and defective spinel structures.



(a) (b)

Figure 2. Isothermal cross-section of the Li–Mn–O phase diagram: (a) – general view of the isothermal section showing phase regions of Li–Mn–O compounds; (b) – enlarged fragment illustrating the location of stoichiometric compositions with rock salt, spinel, and defective spinel structures [3]

The Li-Mn-O system is a complex phase diagram with various stoichiometric and defective spinel structures. The $Mn_3O_4·Li_4Mn_5O_{12}$·λ-MnO_2 triangle characterises these spinel compositions (Figure 2). The general formula $Laxman_3$–xO_4 ($0 \leq x \leq 1.33$) describes stoichiometric spinel structures located between Mn_3O_4 ($x = 0$) and $Li_4Mn_5O_{12}$ ($x = 1.33$). Manganese oxides with the Mn_3–xO_4 ($0 \leq x \leq 1$) defective spinel structure are located between Mn_3O_4 and γ-MnO_2.. The lithiated manganese oxides with the defective spinel structure $Li_2O·yMnO_2$ ($y > 2.5$) are located on the line between $Li_4Mn_5O_{12}$ and λ-MnO_2, which determines the constant valence of the Mn^{4+} ion. Phases with the structure of rock salt $LizMn_3$–zO_3 ($0 \leq z \leq 2$) are located on the line between MnO ($z = 0$) and Li_2MnO_3 ($z = 2$). The $LiMn_2O_4$ compound initially attracted attention as a cathode material for lithium-ion batteries (LIB), being cheaper and less toxic compared to $LiCoO_2$ [4].

The crystal structure of $LiMn_2O_4$ spinel is a tightly packed cubic anionic lattice, where cations occupy octahedral and tetrahedral positions (Figure 3).

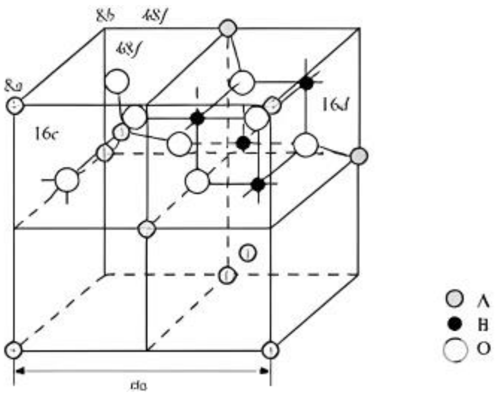


Figure 3. Schematic representation of the spinel structure of type $A[B_2]O_4$ [5]

There are two limiting cation placement options: In normal spinels, A cations are in tetrahedral positions (8a), and B cations are in octahedral positions (16d). Half of the A and B cations in reversed spinels occupy octahedral positions, while the remaining B cations occupy tetrahedral positions. A continuous series of intermediate structures is possible between these variants, allowing cations with different valences to be in various positions. However, one of the disadvantages of $LiMn_2O_4$ is the potential structural rearrangement of spinel into a layered rhombohedral $LiMnO_2$ structure.

One of the disadvantages of $LiMn_2O_4$ as a cathode material for lithium-ion batteries is its relatively low theoretical discharge capacity of 148 mAh/g. At the same time, in practice, this compound can only provide 100-140 mAh/g. On the other hand, lithium nickelate ($LiNiO_2$) was considered a promising cathode material capable of producing about 200 mAh/g. However, its production is fraught with several problems [6].

The $LiNiO_2$ structure, similar to $LiCoO_2$, has a specific setup known as the R3m space group, where lithium and nickel ions are found in octahedral positions 3(a) and 3(b) within a tightly packed cubic arrangement of oxygen. However, there is a fundamental difference between these compounds. The Jahn-Teller effect causes the NiO_6 octahedra to change shape in the nickelate structure because of the Ni^{3+} ions. Also, getting a consistent and correct $LiNiO_2$ composition is difficult because trivalent nickel becomes unstable at high temperatures during the synthesis [7]. Divalent nickel ions take up some of the spots where lithium would generally be in the oxide structure, so the nickelate formula is better expressed as $[Li_{1-z}Ni_z^{2+}]3a[Ni_{1-z}^{3+}Ni_z^{2+}]\ 3b[O_2]\ 6e$. So, for Li_xNiO_2, the structure changes from rhombohedral to monoclinic when the value of x is between 0.4 and 0.75 because of Jahn-Teller distortions and some nickel ions moving to where lithium ions are; at the same time, the rhombohedral structure is present when x is between 0.75 and 1. Also, lithium nickelate breaks down quickly because of reactions with the electrolyte and isn't stable enough when heated, which has restricted its use as a cathode material in industry [8].

Nickel-based compounds show high discharge capacity, like $LiMO_2$-type layered structures with M being Ni, Co, and Mn. Consequently, numerous studies are devoted to creating cathode materials containing nickel-doped cobalt, manganese, and other metals, which can be easily synthesised and have favourable electrochemical characteristics [9].

The presence of manganese, cobalt, and nickel enhances the thermal stability and structural stabilisation of cathode materials. Based on the study of individual oxides $LiCoO_2$, $LiNiO_2$, and $LiMnO_2$, researchers have developed mixed systems combining oxides of cobalt, nickel, and manganese [10].

Since $LiCoO_2$ and $LiNiO_2$ structures belong to the same spatial group, oxides in the Li-Co-Ni-O system can form almost ideal solid solutions [11]. The $LiNi_yCo_{1-y}O_2$ solid solution was proposed and patented in 1989 as a cathode material. Many compounds containing cobalt and nickel have been studied, including $LiNi_yCo_{1-y}O_2$ ($0 \leq y \leq 1$). These metals can synthesize solid solutions in a wide temperature range (500-1000°C). Solid solutions in this system can exist as a layered or spinel structure, depending on the synthesis conditions.

The structure of solid solutions based on $LiNiO_2$ and $LiCoO_2$ is more resistant to lithium deintercalation up to $x = 0.3$ (O_3 type structure), allowing higher capacitance characteristics [12]. However, LixNiyCo$_1$–yO$_2$ compounds are also characterised by a lower degree of cationic mixing than $LiNiO_2$, with an increase in nickel concentration in LixNiyCo$_1$–yO$_2$ oxides, the degree of cationic mixing increases, which is associated with the appearance of divalent nickel. Nevertheless, an increase in the nickel content leads to a rise in the specific capacity of solid solutions in the Li–Co–Ni–O system. LiNiyCo$_1$–yO$_2$ compositions with y = 0.7-0.8 are the most effective [13].

Many scientists have studied the various compositions in this system using multiple synthesis methods, such as solid-phase, coprecipitation, variations of the sol-gel process, and others. According to the data analysis, the conditions and method of synthesis significantly impact the size of the formed particles, morphology, texture, density, and final electrochemical properties of the compounds. In addition, studies have been conducted on compounds in the Li-Ni-Co-O system doped with other elements [14].

Some researchers believe that the compound $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ with an exact ratio of components is the most balanced solid solution composition in the LiCoNiMnO system since it has an optimal combination of capacity, power, lifetime, thermal stability, safety and cost [15]. Numerous works devoted to studying this compound continue to arouse scientific interest. Figure 4 shows the model of the crystalline structure of the $LiNi_1/_3Co_1/_3Mn_1/_3O_2$ layered oxide, illustrating the arrangement of transition metal ions and lithium within the layered framework.

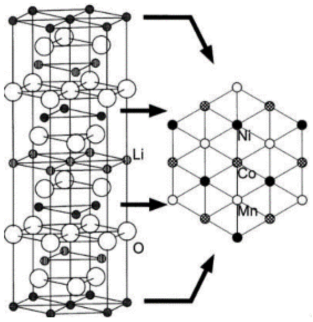


Figure 4. Model of the crystalline structure of the LiNi1/3Co$_1$/$_3$Mn$_1$/$_3$O$_2$ oxide [16]

In addition to the above-mentioned compound $LiNi_1/_3Co_1/_3Mn_1/_3O_2$, there are other compounds in the triad of transition metals nickel, cobalt and manganese that are of industrial interest for use as cathode materials for lithium-ion batteries [17]. The phase diagram of the $LiCoO_2$ - $LiNiO_2$ - $LiMnO_2$ system (Figure 5) shows the compositions used in the industrial production of cathodes for lithium-ion batteries. These compositions are characterised by an optimal combination of electrochemical, structural, and economic parameters, which makes them promising for commercial use as cathode materials.

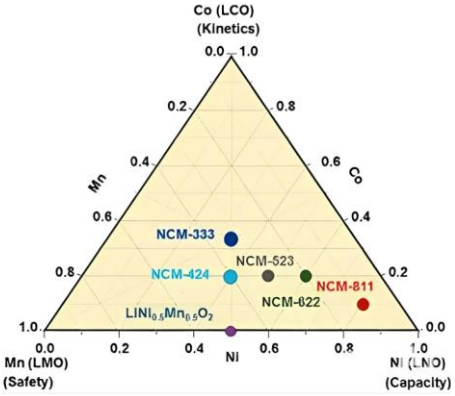
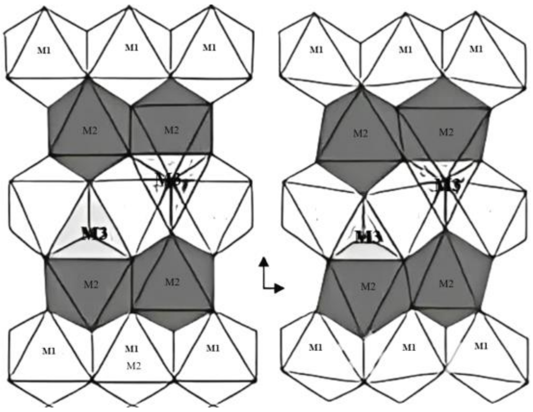


Figure 5. Phase diagram in the triple CO, LMO, LMO system (dots indicate compositions used on an industrial scale [18])

Unlike manganese- and nickel-based cathode materials, lithium iron phosphate ($LiFePO_4$) electrodes with a triphylite structure are characterised by increased structural and thermal stability during lithium ion extraction.

The crystal structure of $LiFePO_4$ belongs to the orthorhombic spatial group Pnma with unit cell parameters a = 1.033 nm, b = 0.601 nm, and c = 0.4693 nm. Figure 6 schematically shows the ideal and real crystal structures of $LiFePO_4$.



(a) (b)

Figure 6. Schematic representation of ideal (a) and real (b) $LiFePO_4$ structures [17]

The $LiFePO_4$ framework structures consist of FeO_6 octahedra and PO_4 tetrahedra touching oxygen vertices in the b-c planes and common edges. The PO_4 tetrahedra are not interconnected. Along the c axis, lithium atoms are located in interstices, forming chains moving along the a-c planes. In the a-c planes, iron atoms form zigzag chains at the vertices of octahedra oriented parallel to the c axis. The iron atoms occupy the M2 positions, and the lithium atoms occupy the M1 positions. The oxygen structure is a slightly distorted, tightly packed hexagonal structure. Due to the strong covalent P-O bonds forming a three-dimensional delocalised chemical bond system, $LiFePO_4$ retains thermodynamic stability even at temperatures up to 200°C.

Significant efforts by researchers are aimed at improving the electrochemical characteristics of existing cathode materials for lithium-ion batteries. Because the breakdown of cathode materials with reactive nickel starts on their surfaces, creating protective coatings is a key research focus. To enhance how well cathode materials work, researchers commonly use methods like applying coatings, making composites, adding different elements, creating core-shell structures, and other ways to modify them.

Covering the surface of cathode materials makes them more stable and enhances their electrical features, like capacitance, how long they last, and speed. Composite materials made of a cathode compound and conductive additives help boost electronic conductivity and enhance electrochemical features. Doping cathode materials with various elements can modify their structure and redox properties and, as a result, lead to a change in electrochemical behaviour. Core-shell structures are also a promising approach for increasing the stability and improving the characteristics of cathode materials [19, 20].

3. Results and discussion

3.1. Lithium-enriched transition metal oxides with the general formula xLi_2MnO_3 (1-x) $LiMO_2$, (M = Mn, Ni, Co)

A promising direction in the field of cathode materials for lithium-ion batteries is complex lithium oxides of nickel, cobalt and manganese with an excess of lithium compared to the stoichiometry of $LiMO_2$, called "Li-rich" in English literature. These materials were first proposed by a group of researchers led by Michael Thackeray from Argonne National Laboratory [21]. Their structure typically includes two compounds: lithium manganate (Li_2MnO_3), which has a monoclinic shape, and lithium oxide ($LiMO_2$), which has a trigonal shape. The formula of these materials can be represented as $xLi_2MnO_3 \cdot (1\text{-}x)LiMO_2$, where $M=Mn_xNi_yCo_z$, x+y+z=1, or formally as $Li_{(1+y)}M'_{(1-y)}O_2$ or $Li[Li_yM'_{(1-y)}]O_2$ in excess of lithium.

The main benefits of Li-rich oxides are their ability to store much energy (over 250 mAh/g) compared to other known materials like $LiCoO_2$ (140 mAh/g), $LiMn_2O_4$ (120-140 mAh/g), $LiNixCoyMnzO_2$ (160 mAh/g), and $LiFePO_4$ (160-170 mAh/g), as well as their high energy output (more than 950 Wh/kg) at an average voltage of 3.5-3.6 V. Additional advantages are their relatively low cost and toxicity due to the reduced content of cobalt and manganese [21].

The Li-rich oxides of the Li_2MnO_3 fraction (monoclinic, pr. C2/m) and $LiMO_2$ (trigonal, pr. R3m) have densely packed face-centred cubic layers with an interlayer distance of about 4.7 Å between the planes (001) in the monoclinic structure of Li_2MnO_3 and (003) in the trigonal structure of $LiMO_2$, which contributes to their interaction.

According to some researchers, the Li_2MnO_3 compound is electrochemically inactive when cycled to a voltage of 4.4 V, which stabilises the structure of the entire material. When cycling to a higher voltage (4.6-4.8 V), a significant increase in the specific capacity of materials is associated with the activation of the monoclinic structure by removing lithium and oxygen, as well as the participation of the oxygen anion $(O_2^- O_2n^-)$ in the redox process. Deeper oxidation and reduction of transition metal cations are also possible. The literature offers various explanations for the abnormally high capacity and the observed decrease in capacity during cycling of Li-rich oxides. Figure 7 presents a schematic comparison of the crystal structures of the monoclinic Li_2MnO_3 phase and the trigonal $LiMO_2$ phase, highlighting their structural differences and layered arrangements.

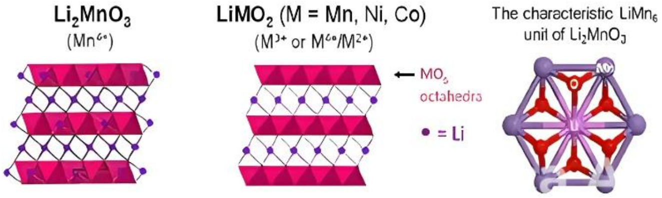


Figure 7. Schematic representation of the structures of the monoclinic phase (Li_2MnO_3) and the trigonal phase ($LiMO_2$) [22]

The key problems associated with using lithium-enriched oxide cathode materials are the reduction of voltage and capacity during cycling, high irreversible capacity on the first cycle, and limited speed characteristics. Despite significant research efforts, no materials that are free of these shortcomings have been found. The reasons for this behaviour of these complex systems have not been definitively established [23]. The formation of spinel-like domains inside the layered phase and side reactions of the surface with the electrolyte and its decomposition products are considered to be one of the factors causing a drop in capacity during cycling. In this case, releasing Li_2O with the formation of oxygen, lithium hydroxide, and carbonate plays a crucial role [24].

As mentioned before, when lithium-enriched oxides are used at high voltage, they change their structure, but we don't fully understand how this happens. The literature emphasises the importance of the initial structure of these materials, but the relationship between the microstructure and its electrochemical properties is currently not completely clear [25]. It also remains unclear whether the lithium-enriched structures are a composite or a solid solution at the nanodomain level. Recent studies using high-resolution transmission electron microscopy and characteristic electron energy loss spectroscopy confirm the existence of a nanodomain structure [26]. The conditions and methods of material synthesis, as well as their composition, are undoubtedly crucial in this matter.

4. Conclusions

In conclusion, it should be noted that the further development of lithium-ion batteries requires close attention to the materials of the positive electrode, particularly to promising lithium-enriched oxides as potential cathode materials. However, the existing disadvantages of these materials, such as limited energy consumption, degradation during cycling, and the complexity of analysing their structures, make their industrial application difficult.

The inconsistency of the data presented in the literature highlights the need for further fundamental research to understand better the influence of various factors on the functional characteristics and performance of lithium-enriched oxides. Identifying ways to improve their performance while reducing degradation during cycling is also necessary. Solving these problems is an essential prerequisite for the possible industrial introduction of lithium-enriched oxide materials as high-energy cathodes for lithium-ion batteries.

Author contributions

Conceptualization: MD; Data curation: AK; Formal analysis: MD; Funding acquisition: AK, SY; Investigation: SY; Methodology: MD; Project administration: XX, XX; Resources: AK, SY; Software: AK, SD; Supervision: MD, AK; Validation: SY; Visualization: AK, SY; Writing – original draft: AK, SY; Writing – review & editing: SY. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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Литий-ионды аккумуляторларға арналған оксидті электрод материалдарына шолу

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Аңдатпа. Мақалада литий-ионды аккумуляторлардың (ЛИА) даму перспективалары қарастырылады, атап айтқанда, катодты материалдар ретінде қолданылатын литийге бай ауыспалы металл оксидтеріне баса назар аударылады. Негізгі назар $xLi_2MnO_3 \cdot (1{-}x)LiMO_2$ (мұндағы M = Mn, Ni, Co) формулалы материалдарға аударылған, олар жоғары разрядтық сыйымдылықпен (250 мА·сағ/г-дан астам) және меншікті энергиямен (950 Вт·сағ/кг-нан жоғары) ерекшеленеді, бұл оларды дәстүрлі катодты материалдар — $LiCoO_2$, $LiMn_2O_4$ және $LiFePO_4$-пен салыстырғанда тиімдірек етеді. Бұл оксидтер Li_2MnO_3-тің моноклиндік фазасы мен $LiMO_2$-нің тригональды фазасын біріктіріп, жоғары өнімділікке қол жеткізеді. Алайда, авторлар бірқатар мәселелерге назар аударады: төмен жылдамдық сипаттамалары, алғашқы циклдағы қайтымсыз сыйымдылық және циклдік жұмыс барысында кернеу мен сыйымдылықтың төмендеуі. Бұл кемшіліктер шпинель тәрізді құрылымдардың түзілуімен, электролитпен шекарада жанама реакциялармен және оттектің бөлінуімен байланысты. Мұндай кемшіліктерді жою үшін қорғаныс жабындарын қолдану, легирлеу және композиттік құрылымдар жасау сияқты модификациялау әдістері ұсынылады. Мақалада сондай-ақ кеңінен қолданылатын басқа катодты материалдарға — $LiCoO_2$, $LiMn_2O_4$, $LiNiO_2$ және олардың комбинацияларына шолу жасалып, олардың артықшылықтары мен шектеулері талданады. Әсіресе, $LiNi1/3Co1/3Mn1/3O_2$ және $LiFePO_4$ сияқты перспективалы материалдарға ерекше көңіл бөлінген, себебі олар электрохимиялық және экономикалық тұрғыдан оңтайлы қасиеттерге ие. Сондай-ақ литийге бай оксидтердің құрылымын оңтайландыру және деградация механизмдерін тереңірек түсіну үшін қосымша зерттеулердің қажеттілігі атап өтілген. Бұл мәселелерді шешу ЛИА үшін жоғары тиімді әрі тұрақты катодты материалдарды әзірлеуге мүмкіндік беріп, электромобильділік пен басқа да энергияны көп қажет ететін технологиялардың дамуына ықпал етуі мүмкін.

Негізгі сөздер: *литий-ионды аккумуляторлар, катодты материалдар, литиймен байытылған оксидтер, Lerich оксидтері.*

Обзор по оксидным электродным материалам для литий-ионных аккумуляторов

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Аннотация. В статье рассматриваются перспективы развития литий-ионных аккумуляторов с акцентом на литий-обогащённые оксиды переходных металлов, используемых в качестве катодных материалов для литий-ионных аккумуляторов (ЛИА). Основное внимание уделяется материалам с формулой $xLi_2MnO_3 \cdot (1{-}x)LiMO_2$ (где M=Mn, Ni, Co), которые демонстрируют высокую разрядную ёмкость (более 250 мА·ч/г) и удельную энергию (свыше 950 Вт·ч/кг), превосходя традиционные катодные материалы, такие как $LiCoO_2$, $LiMn_2O_4$ и $LiFePO_4$. Эти оксиды сочетают в себе моноклинную фазу Li_2MnO_3 и тригональную фазу $LiMO_2$, что обеспечивает их высокую производительность. днако авторы отмечают ряд проблем, включая низкие скоростные характеристики, необратимую ёмкость первого цикла и деградацию напряжения и ёмкости при циклировании. Эти недостатки связаны с образованием шпинелеподобных структур, побочными реакциями на границе с электролитом и выделением кислорода. Для улучшения характеристик предлагаются методы модификации, такие как защитные покрытия, легирование и создание композитных структур. В статья также включен обзор других распространённых катодных материалов, таких как $LiCoO_2$, $LiMn_2O_4$, $LiNiO_2$ и их комбинации, подчёркивая их преимущества и ограничения. Особое внимание уделяется перспективным материалам, включая $LiNi1/3Co1/3Mn1/3O_2$ и $LiFePO_4$, которые обладают сбалансированными электрохимическими и экономическими свойствами. Также было подчёркнута необходимость дальнейших исследований для понимания механизмов деградации и оптимизации структуры литий-обогащённых оксидов. Решение этих задач может способствовать разработке высокоэффективных и стабильных катодных материалов для ЛИА, что важно для развития электромобильности и других энергоёмких технологий.

Ключевые слова: *литий-ионные аккумуляторы, катодные материалы, обогащенные литием оксиды, Lerich оксиды.*

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