Proceedings of CONF-MSS 2026 Symposium: Advanced Composite Materials and Polymer Chemistry
DOI: 10.54254/2755-2721/2026.AD32213

An Overview of Modification Strategies for Ternary Cathode Materials in Lithium-Ion Batteries

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Abstract. The need for lithium-ion batteries with high energy density and long cycle life is becoming more and more urgent. This is because electric vehicles and large-scale energy storage systems are expanding at a rapid speed. Among all the possible positive electrode materials, ternary layered oxides ($LiNixCoyMnzO_2$, or NCM) are seen as a key material system for next-generation high-energy batteries because of their high specific capacity and relatively low cost. But there are some challenges that stand in the way of their real application, such as complicated synthesis processes, unstable structure in high-nickel compositions and serious side reactions at the material interface. This paper gives a detailed and systematic review of the latest progress in the creative synthesis methods and performance optimization ways for NCM positive electrode materials. It also talks about the main preparation methods, including the sol-gel method, coprecipitation and hydrothermal synthesis. The paper sums up the newest development of these techniques, with a focus on making synthesis processes simpler, making materials more homogeneous, controlling the microstructure well and making the electrochemical performance better. In short, new ideas and changes in synthesis routes can make the elemental uniformity, structural integrity and electrochemical stability of NCM materials much better, and in this way, they provide reliable material solutions for the development of high-performance lithium-ion batteries.

Keywords: lithium-ion batteries, cathode materials, ternary materials, modification strategies

1. Introduction

The growing global need for energy and the rising awareness of environmental protection have made it a key research priority to develop effective and green energy storage technologies. Lithium-ion batteries (LIBs) were first made for commercial use by Sony in 1991, and they are now necessary in portable electronics, electric vehicles and power grid energy storage [1]. The reason is that they have high energy density, a long cycle life, no memory effect and are friendly to the natural environment. We can clearly see their growing popularity from the wide use of electric vehicles: the global number of electric vehicles has risen by 63% over the past ten years and reached about 5 million in 2018. Some forecasts also say that electric vehicles will take up 11% to 28% of all the vehicles around the world by 2040. The LIBs that are used in these electric vehicles are designed to work for about 120,000 to 240,000 kilometers. These clear trends show that LIBs have developed into a widely used and sustainable energy technology in the world [2].

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The simple basic components of a lithium-ion battery include the cathode, anode, electrolyte and separator, and these four parts form the core structure of the battery. Cathode materials are of different types, from the common transition metal oxides to the new sulfur and air cathodes, while anode materials are usually made of carbon in most cases. Electrolytes can be in three different forms: liquid, solid or gel, and all of these forms contain lithium salts mixed in suitable solvents. The separator is often made of polymer materials, and it stops the electrodes from touching each other while it still allows ions to pass through it. Metal foil current collectors, such as aluminum (Al) and copper (Cu) foils, are used to collect and transport electric charge in the battery [3,4]. Among all these components, the cathode material is the most important one, because it directly decides the overall using effect and the cost of the battery. This fact also underscores that doing research on cathode materials is of critical importance for battery development.

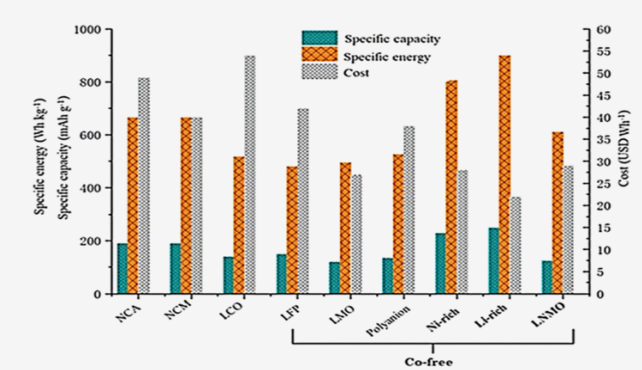


Figure 1. Comparison of cathode materials [5]

Right now, the most developed positive electrode materials for batteries with high energy density include traditional layered transition metal (TM) oxides like $LiMO_2$ (M = Mn, Ni, Co and so on), spinel $LiMn_2O_4$, olivine $LiFePO_4$ and rechargeable lithium oxide systems. Different positive electrode materials have different levels of energy density, production costs and electric capacities, and this specific feature is shown clearly in Figure 1. For lithium-ion batteries that use different kinds of positive electrode materials, people have developed lithium nickel cobalt manganese oxides (lithium nickel cobalt manganese oxides, LNCMO) to apply them in battery production and practical use. These materials mix together $LiNiO_2$, $LiCoO_2$ and $LiMnO_2$ compounds. They take in the unique good points of each single component, and this way can make the whole electrochemical performance of the materials much better. Among all the different types of LNCMO material compositions, $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ (NCM) has been studied in a great deal of detail. The main reasons for this in-depth study are its high reversible capacity, quite low production cost and better thermal stability [6].

Because of these above points, this paper looks over and summarizes the latest research progress on lithium-ion battery positive electrode materials, and it pays special attention to NCM ternary materials. The paper puts main focus on modification methods, electrochemical performance, typical application cases of the materials, and also the existing problems and future development directions for them. It does all these things to offer useful reference materials and practical ideas for the further research and real application of lithium-ion battery positive electrode materials in related fields.

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2. Preparation methods for NCM materials

At present, a variety of methods have been developed for the synthesis of single-crystal NCM cathode materials. Depending on the mixing sequence of transition metals and the physical state of the precursors, these methods can be classified into solid-state reactions, coprecipitation, sol–gel synthesis, spray pyrolysis, template-assisted methods, and others. The synthesis route has a pronounced influence on the morphology, crystallinity, and electrochemical performance of NCM cathode materials.

2.1. Sol–Gel method

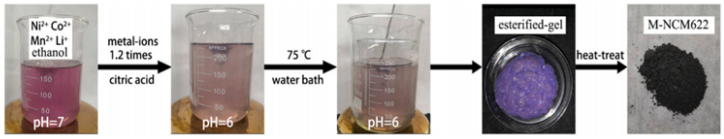


Figure 2. Laboratory-scale preparation process of $Li(Ni_{0.6}Co_{0.2}Mn_{0.2})O_2$ [7]

The latest lab-scale sol-gel synthesis for NCM cathodes is shown in Figure 2. In standard methods, deionized water acts as the solvent. Gel formation requires a large chelating agent dose of citric acid and continuous pH regulation via aqueous ammonia addition. The citric acid dosage is usually set at twice the total metal ion molar amount. This ensures full metal ion chelation while the excess acid acts as a matrix for polymer gel network development. However, high acidity from excess acid inhibits the forward dissociation of citric acid. Therefore, aqueous ammonia is continuously supplied to neutralize H^+, activating the chelating function and driving citrate ion production. During water evaporation at 95 °C, the extra citric acid condenses and bridges with metal-citrate complexes, forming the final 3D network structured polymeric chelate gel [7].

The effectiveness of this method is offset by its high citric acid demand and the need for continuous pH adjustment with aqueous ammonia, imposing stringent operating conditions. Although it yields NCM cathodes with excellent electrochemical performance, process complexity hinders large-scale industrial adoption. However, in 2021, Zhu Jiping and co-workers reported an innovative sol-gel method based on a new mechanism—the esterification of metal-citrate complexes with ethanol to form a gel spontaneously. This method's main improvements are the removal of both excess chelating agent and the cumbersome real-time pH control, dramatically simplifying the process and reducing experimental difficulty. Here, ethanol is the reaction medium, with gelation driven by esterification between residual –COOH on the complexes and ethanol's –OH groups. The authors further proposed that a citric acid-to-metal ion molar ratio of 1.2:1 maintains an appropriate pH, facilitating smooth esterification and successful gelation [7].

Table 1. EDS elemental ratios, lattice parameters, and XRD peak intensity ratios of modified NCM (M-NCM622) and traditional NCM (T-NCM622) [7]

Example	EDS elemental ratio	a(nm)	c(nm)	c/a	I(003) /I(104)
M-NCM622	Li(Ni0.58Co0.21Mn0.21)O2	0.287646	1.421436	4.9416	0.97
T-NCM622	Li(Ni0.54Co0.23Mn0.24)O2	0.288761	1.424951	4.9347	0.87

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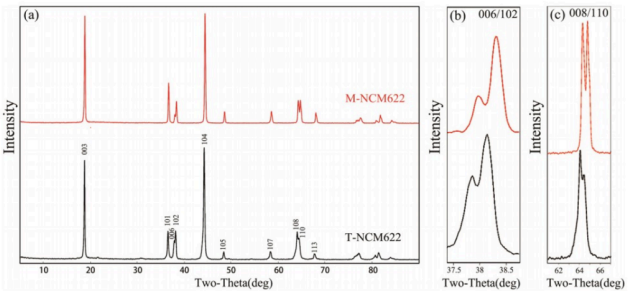


Figure 3. (a) XRD patterns of modified NCM (M-NCM622) and traditional NCM (T-NCM622), and enlarged diffraction peaks of (b) (006)/(102) and (c) (008)/(110) [7]

EDS semi-quantitative test in Table 1 shows that the content of Ni, Co and Mn in M-NCM622, which is made by the modified sol-gel method, is more close to the theoretical 6:2:2 ratio than that in T-NCM622. And this fact means that the metal ions in the esterified gel are distributed in a much more even way. Both of the two samples have clear XRD peaks as shown in Figure 3, these peaks match the hexagonal α-$NaFeO_2$ structure (R-3m space group) and there are no impurity phases in either sample. This result confirms that both samples have high crystallinity and high purity. M-NCM622 also has a regular morphology, and its particles are larger and gather closely together with the size about 400 nanometers. But T-NCM622 has an irregular morphology, its particle size has a wide range from 200 to 500 nanometers and its structure is loose with many small pores[7]. And in line with this, the modified material has better electrochemical performance: it has a high initial discharge capacity of 223.7 $mA \cdot h \cdot g^{-1}$ at the current rate of 0.1 C, and it also has stable cycling performance. 116.8 $mA \cdot h \cdot g^{-1}$ of capacity is retained after 50 cycles at 1 C, with the capacity retention rate reaching 80.6% [7]. So this improved sol-gel method can successfully produce high-performance NCM cathode materials, and it also relieves the traditional problems like complex operation steps and overuse of chemical reagents. This method thus becomes a main way for synthesizing single-crystal NCM cathode materials.

2.2. Coprecipitation method

Because the purity of the final NCM material directly determines the electrochemical performance of the resulting battery, homogeneous powder precursors—such as mixed hydroxides or double hydroxides—are regarded as a prerequisite. Among the available approaches, the coprecipitation method is considered one of the most effective routes for preparing metal hydroxide precursors, as it can generally yield oxide products with high phase purity [8]. In 2004, Lee and co-workers demonstrated through systematic experiments that precise control of pH, chelating agent concentration, and stirring speed are three critical factors for obtaining materials with a narrow particle size distribution and high tap density during coprecipitation. When the pH value was varied from 11 to 12, the particle size decreased from approximately 10 μm with relatively broad size distribution at pH = 11 to smaller, more uniform particles with a quasi-spherical morphology at pH = 12. Moreover, with increasing NH_3^+ concentration, uniformly spherical particles were formed and the particle size distribution became narrower. The effect of stirring speed is mainly reflected in the material density: higher stirring speeds generally lead to higher packing density of the obtained materials [9].

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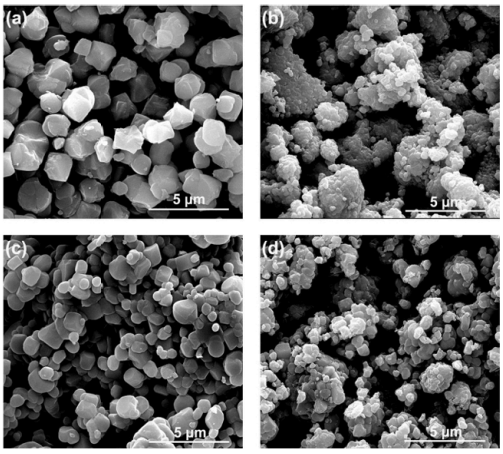


Figure 4. SEM images of the oxalate precursor (a), and NCM-622 powders calcined at 800 °C (b), 850 °C (c), and 900 °C (d)

Yang et al. (2014) enhanced the synthesis process via a distribution strategy, using a two-step mixed suspension evaporation to obtain a precursor. The thermal treatment carried out later has successfully made 1D hierarchical $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ micro-rods (called NCM-MRs for short), and these micro-rods are assembled from tiny nanoparticles. When compared with micron-sized particles made by the traditional coprecipitation method, the NCM-MRs show much better coulombic efficiency, discharge capacity, rate performance and cycling stability. Within the voltage range of 2.8 to 4.3 volts, they can release an initial capacity of 163.3 $mA \cdot h \cdot g^{-1}$ at the current rate of 0.1 C, with the coulombic efficiency reaching 92.0%. They can keep a capacity of 135.3 $mA \cdot h \cdot g^{-1}$ at 5 C and 126.9 $mA \cdot h \cdot g^{-1}$ at 10 C respectively [10].

In 2019, Yao and his research team created a new oxalate coprecipitation method (OCP for short). They use oxalic acid as a precipitating agent that is cheaper and more eco-friendly than inorganic alkalis, and this agent can still keep the materials mixed at the atomic level. The samples heated at 800, 850 and 900 degrees Celsius were named S800, S850 and S900 (see SEM/Fig.4 and XRD/Fig.5). After 100 charge and discharge cycles at the current rate of 1 C, S850 kept the highest residual capacity of 152 $mA \cdot h \cdot g^{-1}$, with the retention rate at 93.2%. This number is far higher than that of S800 (77.5%) and S900 (66.5%), as the capacity of these two samples dropped to about 100 $mA \cdot h \cdot g^{-1}$. People usually hold that this kind of capacity loss is caused by bad changes on the material surface and the decomposition of electrolyte, which are induced by Ni^{4+} ions and residual lithium. S850 has better stability because its layered structure is improved and it shows little cation mixing. It also has much better rate performance, reaching 76.8 $mA \cdot h \cdot g^{-1}$ at 10 C with clear discharge plateaus, and its capacity can fully recover to 170 $mA \cdot h \cdot g^{-1}$ at the current rate of 0.2 C. So the OCP method can provide a low-cost and easy-to-control way to make multicomponent layered cathode materials [11].

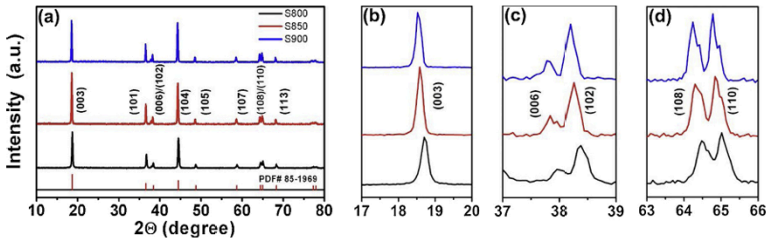


Figure 5. XRD patterns of NCM powders calcined at different temperatures

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In 2022, Lee and his research team improved the coprecipitation method by using a Taylor–Couette reactor. The research work they did showed a key fact: the temperature in the process of precursor synthesis is a vital factor that influences the particle morphology of the precursors and the final cathode materials, including both primary and secondary particle shapes. People find that a lower temperature in the coprecipitation process can improve the cycle life and rate performance of the materials, and it can also raise the Li^+ diffusion coefficient and the exchange current density at the same time. This research brings new ideas for producing high-nickel NCM cathode materials at low temperatures, and it also makes the preparation process of precursors at different temperatures much clearer [12]. In short, the single-crystal $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$ made by the coprecipitation method has extremely good dispersion and electrochemical performance. This method needs people to control the centrifugation and feeding rates in a precise way, and it also requires an additional lithiation step after the main process. Even so, it is a very promising method and has great long-term development potential for the research and production of high-performance NCM cathode materials.

2.3. Hydrothermal synthesis method

The hydrothermal synthesis method is a fairly new approach to make NCM cathode materials, and it has not been applied for a long time. This method is a further development and improvement of the coprecipitation method in fact. In most hydrothermal synthesis processes, people first prepare the reaction precursors by using the coprecipitation method. Then they conduct hydrothermal treatment to get the typical layered NCM materials we need. A major advantage of hydrothermal reactions is that they can effectively prevent the precursor from dissolving and recrystallizing in the whole reaction process. Also, the reaction temperature of hydrothermal method is relatively low, which makes the final calcination temperature lower as well. In this way, it becomes possible to synthesize NCM materials with high purity. In 2005, Myung successfully made layered $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ with the hydrothermal method. He hydrothermally treated the spherical $(Ni_{1/3}Co_{1/3}Mn_{1/3})(OH)_2$ made by coprecipitation in a LiOH aqueous solution with excess LiOH at the temperature of 170 °C. The product prepared directly in this way had relatively low crystallinity, and its discharge capacity was also limited. But it still showed quite good reversibility in the electrochemical reaction. After the follow-up thermal treatment was carried out, the structural integrity of the $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ synthesized by the hydrothermal method was greatly improved. It could achieve a high discharge capacity of 157 $mA{\cdot}h{\cdot}g^{-1}$ when the cutoff voltage was set at 4.3 V, and 182 $mA{\cdot}h{\cdot}g^{-1}$ when the cutoff voltage was 4.6 V. What's more, it kept excellent reversibility all the time during the process [13].

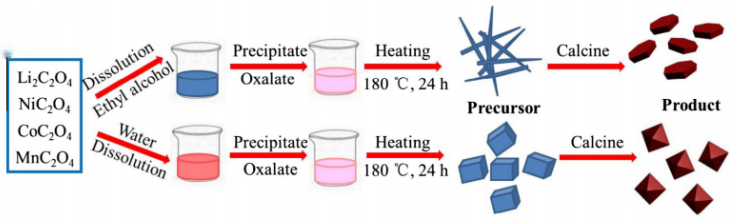


Figure 6. Schematic illustration of the possible formation mechanisms of the precursor and the final product [14]

In 2015, Fu and the research team prepared $Li_{1.2}Mn_{0.56}Ni_{0.12}Co_{0.12}O_2$ (LMNCO) materials with the hydrothermal synthesis method. Their study put main focus on the effect that the solvent has on the products made during the synthesis process. They also compared the charge and discharge

DOI: 10.54254/2755-2721/2026.AD32213

abilities of crystals from different synthesis ways under the same test conditions. They drew a conclusion that the synthesis way plays a key role in crystal growth, shape control and electrochemical performance of the materials. Ethanol and water were each used as the solvent to synthesize LMNCO materials, and this experimental design is shown in Figure 6. The SEM images corresponding to these two kinds of materials are presented in Figure 7. The test results show that both the particle size and the crystal shape are greatly influenced by the solvent that is used in the synthesis. In particular, the LMNCO material made with ethanol as the solvent had much better charge–discharge performance. It achieved a reversible capacity as high as 306.9 $mA \cdot h \cdot g^{-1}$ at the current rate of 0.2 C [14]. This research work expanded the range of solvent choices for hydrothermal synthesis. It also provided useful ideas for the research community to do further exploration in this related field.

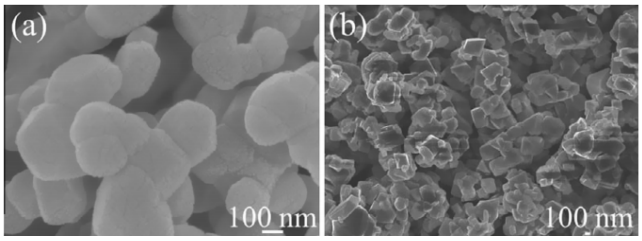


Figure 7. (a) LMNCO synthesized using ethanol as the solvent; (b) LMNCO synthesized using water as the solvent [14]

In 2023, Kang Siyi and his research team made a full and detailed review on hydrothermal synthesis methods. They introduced the processing steps, used equipment and different types of products that can be made by hydrothermal synthesis in a systematic way. They also summed up and discussed the current challenges this synthesis technique is facing and the possible development directions for its future application. Even though a large number of studies have reported hydrothermal synthesis routes for making cathode materials, the authors pointed out that systematic research on the hydrothermal synthesis of NCM cathode materials is still quite limited. Besides, the selection and optimization of surfactants that are used during the hydrothermal synthesis process need more in-depth research and exploration. They also put forward a new point that coating electrode materials and introducing nitrogen or boron doping could be effective ways to improve the working performance of composite materials [15]. This review not only offered a detailed and clear overview of the development process of hydrothermal synthesis, but also provided practical and constructive ideas for solving the existing challenges and promoting the further development of related research work in this field.

3. Conclusions and outlook

This paper has made a systematic review on the latest progress in the preparation of high-nickel layered oxide (NCM) cathode materials and the strategies to optimize their performance. The sol–gel method applies a new reaction mechanism based on esterification. It makes metal ions distribute evenly at the atomic scale, and this effectively improves the structural consistency and lattice integrity of the materials. At the same time, this method greatly simplifies the complex chelation and pH regulation steps in traditional preparation processes. It also shows a good scalability in the experiments carried out at the laboratory level. The coprecipitation method uses stepwise synthesis strategies, new types of precipitating agents and innovative reactor designs. It can control the morphology and composition of precursors in a highly precise way. The materials made by this

DOI: 10.54254/2755-2721/2026.AD32213

method have high purity, high tap density and excellent cycling stability. So this approach is especially suitable for the controllable synthesis and engineering-scale production of high-nickel NCM systems. Hydrothermal synthesis, by contrast, makes crystals grow at a relatively low temperature. It avoids the happening of high-temperature phase transitions and the formation of impurities. This method can produce NCM materials with clear layered structures and high crystallinity. What's more, the flexible choice of solvent systems can further control the product's morphology and electrochemical behavior in an effective way.

Though, as the nickel content in NCM materials increases—especially in high-nickel systems where nickel accounts for more than 80%—their practical application still faces a series of serious challenges. These challenges basically come from the intrinsic structural instability of the materials during the process of electrochemical cycling. Under high-voltage working conditions, deep delithiation easily causes the loss of lattice oxygen and harmful phase transformations. This loss and transformation will lead to a continuous drop in voltage. Lithium ions are inserted into and extracted from the materials repeatedly, which generates anisotropic lattice strain. The strain makes stress build up at the internal defects inside the particles, triggering the formation of microcracks and speeding up the degradation of material performance. At the same time, the more intense side reactions between the highly reactive cathode surface and the electrolyte make the interfacial impedance rise and cause the dissolution of transition metals. What people care about most is that high nickel content makes the thermal stability of the materials much worse, bringing potential risks of thermal runaway. So to make real and substantive breakthroughs in NCM material performance, we need to solve these fundamental scientific problems through cross-scale collaborative design and systematic engineering exploration.

For the future development, NCM cathode materials should follow a step-by-step pathway of "from microscopic mechanisms to macroscopic systems". This pathway will guide the materials to develop toward better performance, higher safety and stronger sustainability. First, at the basic research level, it is extremely essential to deeply combine in situ characterization techniques with multiscale computational simulations. This combination can dynamically explain the structural evolution, ion transport and interfacial reaction mechanisms during cycling at the atomic scale. In this way, it can provide a firm theoretical foundation for the precise design of NCM materials. On this basis, we need to develop an integrated modification strategy of "bulk–interface–structure" for NCM materials. We can stabilize the crystal framework through doping, stop interfacial side reactions by surface coatings and reduce microcrack formation via single-crystal or morphology-optimized particle designs. Doing these things can comprehensively improve the intrinsic properties of NCM materials.

Besides, to meet the different requirements of various application scenarios, the research and development ways for NCM materials must be refined in a corresponding manner. In the field of electric vehicles, the main goals are to achieve higher energy density and faster charging capability. And at the same time, we need to ensure the safety of the materials during their whole service life. This means we need to develop NCM materials toward high-nickel, low-cobalt or even cobalt-free compositions. We also need to make high-voltage-tolerant electrolytes and build stable interfacial architectures. These can support the stable operation and thermal safety of the materials under fast-charging conditions. In large-scale energy storage applications, long cycle life, high safety and low production cost become the main considerations. We can achieve these goals by combining material design and system-level integration. Material design includes moderate nickel content and enhanced structural integrity, while system-level integration covers efficient thermal management and

DOI: 10.54254/2755-2721/2026.AD32213

intelligent state monitoring. This combination can ensure the economic viability and reliability of the materials during long-term and steady-state operation.

Finally, during the entire lifecycle of NCM material development and application, it is necessary to build a comprehensive technological framework that includes green and low-carbon synthesis methods and efficient recycling technologies. We can optimize the synthesis processes to cut down energy consumption and production costs. We can also develop high-efficiency and low-loss recycling and regeneration technologies for NCM materials. In this way, a closed-loop resource cycle can be built in the lithium battery industry. This closed-loop cycle will in turn support the long-term sustainable development of electric vehicles and emerging power systems.

The constant evolution of NCM cathode materials is a systems-level task. It integrates materials science, electrochemistry, engineering technology and systems science together. We can only make high-energy-density, high-safety and long-lifespan NCM batteries play their critical role in transportation electrification and the transformation of global energy systems in the end. And this can only be achieved through vertical integration and coordinated innovation across mechanisms, materials, batteries and different application scenarios.

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