Proceedings of CONF-FMCE 2026 Symposium: Smart City and Infrastructure Engineering
DOI: 10.54254/2755-2721/2026.CH38783

Preparation of Lithium-Rich Manganese-Based Cathode Materials and Their Research in Lithium Batteries

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Abstract. Against the backdrop of the global transition of energy structure toward green and high-efficiency systems, developing sustainable and high-performance lithium-ion battery technology is of great significance. As one of the core components determining the electrochemical performance of batteries, cathode materials for lithium-ion batteries directly affect battery endurance and cost control. Lithium-rich manganese-based cathode materials, with the advantages of high specific discharge capacity and abundant low-cost raw materials, have been regarded as ideal candidates to break the energy density bottleneck of conventional lithium batteries in recent years. Combined with current research hotspots, this paper systematically introduces the basic structure of lithium-ion batteries and the rocking-chair energy storage and conduction mechanism. It focuses on discussing the performance shortcomings of commercial cathode materials including traditional layered oxides, spinel structures and polyanion-type materials. Four preparation methods of lithium-rich manganese-based materials, namely co-precipitation method, high-temperature solid-state method, sol-gel method and hydrothermal method, as well as the electrochemical performance of materials with different micro-morphologies used in lithium-ion batteries are summarized. By comparing key properties such as specific discharge capacity and cyclic retention rate among various cathode materials and lithium-rich manganese-based samples synthesized via different preparation routes, this paper reveals the great application potential and existing technical challenges of lithium-rich manganese-based cathode materials in high specific-energy lithium-ion batteries, providing theoretical support for the sustainable development of high-performance and low-cost lithium-ion batteries.

Keywords: lithium-rich manganese-based, cathode material, lithium battery, performance research, material preparation

1. Introduction

As the concept of sustainable development gains wide acceptance, the global energy crisis and environmental problems become increasingly severe. Developing efficient, clean and sustainable new energy storage and conversion technologies has gradually become a research priority in the energy field. With merits of high energy density, long cycle life and environmental friendliness, lithium batteries are widely recognized as an important technical route to address energy and environmental issues, and serve as one of the key technologies for sustainable energy utilization [1].

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As a critical part governing lithium battery performance, electrode materials' microstructures and electrochemical properties directly influence the overall energy density and output power of lithium batteries. Cathode materials, the key component determining lithium batteries' energy density, cycle stability, safety performance and production cost, have long been the research focus in energy storage materials. Traditional cathode materials such as lithium cobalt oxide, lithium manganate, as well as currently prevailing lithium iron phosphate and nickel-cobalt-manganese ternary materials, possess mature manufacturing techniques and have achieved large-scale industrial mass production. Nevertheless, their low upper limit of theoretical energy density can hardly satisfy the development demand of future long-endurance high-power batteries [2]. High-nickel ternary materials deliver higher discharge capacity, but their fabrication heavily relies on scarce noble metal resources such as nickel and cobalt. Moreover, crystal structure collapse and metal ion dissolution tend to occur during high-voltage cycling [3]. In contrast, lithium-rich manganese-based cathode materials, featuring stable high specific discharge capacity (> 250 mAh/g), high average discharge voltage (> 3.5 V) [4] and low cost, rank among the most promising cathode materials for next-generation lithium batteries [5]. However, such materials suffer from irreversible lithium loss and low initial Coulombic efficiency. Different preparation processes exert remarkable impacts on the crystal structure, micro-morphology and electrochemical performance of materials, and further determine the upper limit of battery capacity. Based on the above research background, this paper systematically sorts out the fundamental working principle of lithium batteries, summarizes and analyzes the synthesis approaches, process optimization and electrochemical performance of lithium-rich manganese-based cathode materials, aiming to offer theoretical references for the research and design of high-performance lithium batteries.

2. Lithium-ion batteries and their energy storage devices

2.1. Brief introduction to lithium-ion batteries

Lithium-ion batteries are the mainstream new-type electrochemical energy storage devices at present. They lie between conventional physical capacitors and other chemical storage batteries, combining the advantages of fast response of capacitors and large-capacity energy storage of storage batteries, and serve as critical energy-storage devices for new-energy vehicles, electronic equipment and other fields. A lithium-ion battery consists of a cathode and an anode, separated by electrolyte containing dissociated salts to enable ion transport between the two electrodes. Once the electrodes are externally connected, chemical reactions proceed simultaneously on both electrodes to release electrons, and electric current becomes available for use, as shown in Figure 1 [6]. Different from capacitors that store charge by physical adsorption, lithium-ion batteries realize energy storage and release through reversible redox reactions between electrodes and electrolyte. Charging and discharging are accomplished with the intercalation and deintercalation of lithium ions between cathode and anode, thus delivering considerable energy density and favorable cycle reversibility. The core working mechanism of lithium-ion batteries relies on the migration, intercalation and deintercalation of lithium ions, which brings fundamental advantages over other chemical systems. First of all, lithium has the lowest reduction potential among all elements, enabling lithium-ion batteries to achieve the highest cell potential. Besides, lithium owns one of the smallest ionic radii among all charged ions. These factors endow lithium-ion batteries with high volumetric capacity. Thanks to numerous merits including stable charge-discharge performance, high energy density, long service life and environmental friendliness, lithium-ion batteries have achieved large-scale commercial application. According to the types of cathode materials, they can be divided into

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lithium iron phosphate, high-nickel ternary, lithium cobalt oxide, lithium-rich manganese-based and other systems.

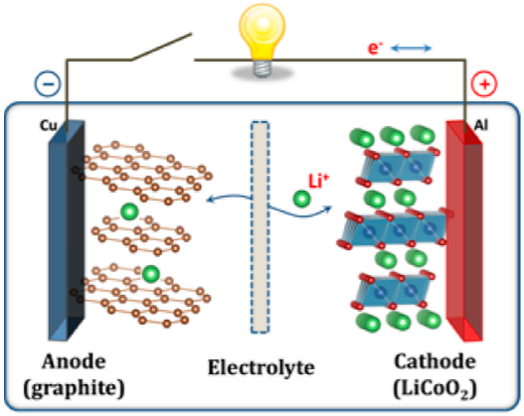


Figure 1. Schematic diagram of lithiumion battery device

2.2. Energy-storage devices of lithium-ion batteries

Lithium-ion batteries are typical electrochemical energy-storage components that can realize the conversion between electric energy and chemical energy. Their energy-storage structure is composed of multi-layers including micro-crystalline particles, cell, housing and control system. A complete cell contains four fundamental components: cathode material, anode material, electrolyte and separator, as illustrated in Figure 2. The energy storage of lithium-ion batteries is realized via redox reactions of cathode and anode materials (rocking-chair mechanism). During charging, lithium ions (Li^+) deintercalate from cathode active substances, migrate through electrolyte and separator toward the anode; the reverse process occurs during discharging when lithium ions return to the cathode. The crystal structure of electrode active materials, high-voltage stability of electrolyte and pore structure of separator exert direct influences on working efficiency, service life and safety performance of batteries.

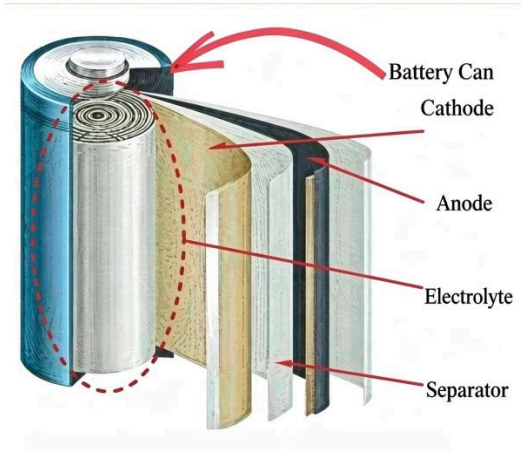


Figure 2. Structure of lithiumion battery for energy storage

3. Conductive mechanism and energy-storage principle of lithium-ion battery devices

Lithium-ion batteries follow the rocking-chair energy-storage and conductive mechanism as a whole. Energy conversion is realized by reversible intercalation, deintercalation and migration of

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lithium ions within the lattices of cathode and anode during battery operation. Driven by external electric field in the charging process, lithium ions deintercalate from the lattices of cathode materials, migrate through electrolyte and separator into anode lattices, while electrons flow through the external circuit. Electric energy is converted into chemical energy for storage. Discharging is the reverse reaction: lithium ions deintercalate from the anode and migrate to the cathode, and electrons move directionally along the external circuit to generate electric current. Chemical energy is converted into electric energy to supply power for external equipment. Relying on two-electrode redox reactions, lithium-ion migration takes place inside the battery, and electron transport forms current outside the battery, achieving stable conversion between electric energy and chemical energy, as shown in Figure 3.

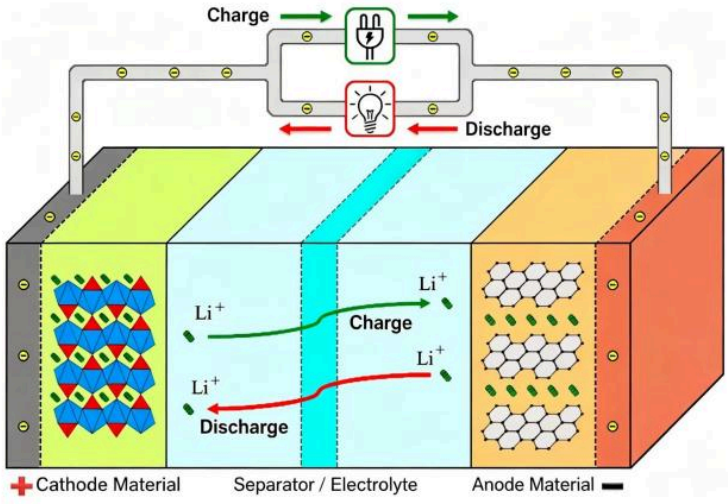


Figure 3. Conductive mechanism and energystorage principle of lithiumion battery

4. Types of cathode materials for lithium-ion batteries

4.1. Layered oxides

Layered oxides include lithium cobalt oxide, high-nickel ternary materials and others. Among them, lithium cobalt oxide ($LiCoO_2$, LCO) is the earliest commercialized layered cathode material. It possesses a layered Li-O-Co crystal structure. Its crystal structure remains stable and the layered structure is well preserved under normal operating voltages, which grants it a longer cycle life. Besides, lithium cobalt oxide powder has a high tap density, facilitating high electrode loading and thus improving energy density. Nevertheless, surface degradation, damages induced by destructive phase transitions and heterogeneous reactions may lead to rapid decay in capacity, efficiency and cycle life [7]. The second category is high-nickel ternary materials (NCM). Studies demonstrate that the core advantage of this material lies in its high energy density. Meanwhile, increased nickel content reduces reliance on expensive cobalt resources, enabling relatively controllable costs. Moreover, certain high-nickel ternary materials can retain nearly 100.0% of their capacity after 100 cycles under specific conditions [8]. However, high-nickel NCM cathode materials tend to generate side reactions at the interface with the electrolyte. Side reactions and their products impair battery performance. In addition, its high energy density brings severe fire hazards, and thermal runaway occurs readily especially under low voltage.

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4.2. Spinel structure

The typical representative of spinel-type lithium-ion cathode materials is lithium manganate (LMO). Lithium manganate ($LiMn_2O_4$) stands among promising cathode materials thanks to its environmental friendliness, low cost and abundant reserves. Furthermore, dual doping with magnesium (Mg) and aluminium (Al) improves the structural stability and high-rate performance of lithium manganate cathode materials. A capacity retention of 84.6% can still be maintained after one thousand cycles at a high rate of 20C [9]. Still, manganese dissolution, electrolyte decomposition and insufficient cycling stability restrict its further applications.

4.3. Polyanionic compounds

One typical representative of polyanionic cathode materials is lithium iron phosphate (LFP). Lithium iron phosphate ($LiFePO_4$) is also one of the most commercially successful cathode materials available today. Existing research shows that innovative wet slurry grading technology can be adopted to adjust particle size and morphology. The modified $LiFePO_4$ delivers high compaction density and large specific discharge capacity [10]. However, its olivine structure, constructed of FeO_6 octahedra and PO_4 tetrahedra, results in poor electronic conductivity. The PO_4 units isolate the connections between FeO_6 groups and block electron transport. Meanwhile, Li^+ ions can only diffuse one-dimensionally through narrow channels, which are prone to blockage.

5. Preparation methods of lithium-rich manganese-based (LRM) cathode materials

Lithium-rich manganese-based materials, with the general formula ($xLi_2MnO_3 \cdot (1-x)LiTMO_2$,TM=Ni,Co,Mn), belong to layered oxide cathode materials. They rely on redox reactions of transition metal cations and lattice oxygen anions. Their theoretical specific capacity can exceed 250 mAh/g, and the energy density reaches 900–1000 Wh/kg. With abundant manganese resources and low cost, they are one of the most promising cathode materials for next-generation high-energy-density lithium-ion batteries. Multiple preparation routes have been developed, including co-precipitation method, high-temperature solid-state method, sol-gel method and hydrothermal method. Distinct differences exist among these techniques in precursor synthesis, processing conditions and product characteristics, as shown in Table 1.

Table 1. Preparation methods of lithium-rich manganese-based materials andproduct features of each process

Preparation Method	Core Process	Key Process Conditions	Advantages of the Process	Disadvantages of the Process	Product Characteristics	References
Co-precipitation method	Divided into overflow method and thickening method; metal salts precipitate to form precursors. After filtration and drying, precursors are mixed with	Oxygen introduction, oxalic acid complexing agent addition, elevated precipitation temperature and prolonged ageing time; pH is raised in overflow method to increase tap density,	High homogeneity of elemental mixing;	Sensitive to reaction conditions; minor fluctuations in pH, temperature, stirring speed may induce obvious changes in precursor morphology and particle size,	Loose and porous precursors can be obtained; porous spherical materials can be fabricated; favorable crystal and layered structures;	[11, 12]

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Table 1. (continued)

Co-precipitation method	lithium sources and calcined in segmented high-temperature steps; acetate ester systems can be adopted for process improvement	while the opposite applies to thickening method; modified process is sintered at 800℃	precursor morphology can be precisely tuned by adjusting reaction conditions	imposing strict requirements on experimental control	uniform particle morphology	[11, 12]
High-temperature solid-state method	Raw materials are ground according to stoichiometric ratios, uniformly mixed with polyvinyl alcohol, calcined in a muffle furnace under air atmosphere and cooled naturally	Ball milling for approximately 4 h; slow heating to roughly 700℃ for calcination; constant temperature holding for 5 h; air atmosphere	Simple process, easily accessible raw materials, low cost, short experimental cycle, suitable for batch preparation	Poor elemental mixing uniformity; difficult to control particle morphology; high calcination temperature and long holding time lead to high energy consumption	Simple process; water-system lithium-rich manganese-based can be obtained	[13]
Sol-gel method	Acetates are dissolved in distilled water. Citric acid and ascorbic acid are added, and ammonia water is used to adjust pH. Water bath evaporation and drying produce gel; pre-calcination removes organics, followed by high-temperature sintering, grinding and sieving	Sintering at 850–900℃ for several hours; products sieved through a 200-mesh sieve	Relatively high elemental mixing uniformity; product particle size adjustable by modifying sol pH and other parameters; controllable stoichiometric ratio and good product quality	High cost of raw chemical reagents; long experimental workflow; only applicable to laboratory synthesis, not for large-scale industrial production	Uniform component mixing; powdered lithium-rich manganese-based materials can be prepared	[14]
Hydrothermal method	Metal acetates are dissolved, urea and glycerol are added for hydrothermal reaction in an autoclave. Water/ethanol washing removes impurities to yield quasi-spherical precursors.	Hydrothermal temperature: 150–220℃;	Material micro-morphology tunable via reaction conditions; relatively low reaction temperature restrains excessive grain growth;	Sealed high-pressure system required, hard to implement for large-scale industrial production;	Quasi-spherical precursors obtained;	[15]

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Table 1. (continued)

Hydrothermal method	Precursors are mixed with lithium source in ethanol system, dried and calcined	different calcination temperatures are used to tailor materials	good crystallinity, few impurities and favorable particle dispersity	sensitive to reaction conditions; high production cost	particle morphology and crystal facets adjustable by changing calcination temperature	[15]

6. Study on electrochemical performance of lithium-rich manganese-based materials for lithium-ion batteries

Lithium-rich manganese-based cathode materials exhibit remarkable advantages in electrochemical performance, as shown in Table 2 (the average discharge voltage ranges from 3.5 to 3.6 V).

Table 2. Study on electrochemical performance of lithium-rich manganese-based materials with different morphologies as cathodes for lithium-ion batteries

Material Morphology	Main Preparation Methods	Initial Specific Discharge Capacity at 0.1C / (mAh/g)	Cycling Performance	Energy Density / (Wh/kg)	References
Spherical	Co-precipitation method, sintering of spherical precursors	260–285	Capacity retention of approximately 88% after 200 cycles at 2C	910–1026	[11, 12]
Rod-like	Co-precipitation combined with high-temperature calcination, oxalate-directed one-dimensional precursors	270–290	Capacity retention of approximately 85% after 100 cycles at 1C	945–1044	[16]
Quasi-spherical	High-temperature solid-state method, modified co-precipitation method, hydrothermal method	250–270	Capacity retention of about 92.1% after 100 cycles at 0.2C and 80.3% after 500 cycles at 1C	875–972	[17]
Powdery	Sol-gel method, conventional high-temperature solid-state method	240–250	Capacity retention of approximately 72.3% after 100 cycles at 1C, which can be further improved after modification and optimization	840–900	[18]

Spherical materials possess loose and porous structures, uniform spherical particles, moderate specific surface area, high tap density and good processability, and deliver outstanding high-rate cycling performance [11, 12]. For rod-like lithium-rich manganese-based materials, short rods are about 2 micrometres in length and 300 nanometres in thickness, while long rods are 4–10 micrometres in length and about 600 nanometres in thickness. They show good dispersion and shortened diffusion distance for lithium ions, leading to superior rate performance [16]. Some quasi-spherical materials have outer shells composed of radially aligned nanorods, which can accelerate the diffusion rate of electrons and lithium ions and reduce internal stress induced by phase transitions [17]. Powdery materials feature irregular powder morphology with a broad particle size distribution (100 nm–5 μm) and large specific surface area. They have high initial capacity, and their electrochemical properties can be further enhanced after modification [18].

Although lithium-rich manganese-based cathode materials currently suffer from low initial Coulombic efficiency, voltage decay upon long cycling and interfacial side reactions triggered by oxygen release, their advantages in specific capacity, capacity retention over hundreds of cycles,

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steady-state Coulombic efficiency and high energy density have established their irreplaceable position in the sustainable development of high-specific-energy batteries when comprehensive performance data are considered.

7. Conclusions

Relying on the redox mechanism of transition metal ions, lithium-rich manganese-based cathode materials can be fabricated into high-energy-density cathodes with stable specific discharge capacity for lithium batteries by tuning micro-morphology through preparation routes including the co-precipitation method, high-temperature solid-state method, sol-gel method and hydrothermal method. Spherical and quasi-spherical morphologies can accelerate lithium-ion diffusion and exhibit excellent capacity retention. Spherical morphology delivers high initial specific discharge capacity, moderate specific surface area, high tap density and favourable processability, suitable for high-rate cycling. Powdery morphology has the largest specific surface area and shows great development potential after modification. Despite the drawbacks of low initial Coulombic efficiency, long-cycle voltage decay and oxygen-release-induced interfacial side reactions, core merits such as abundant manganese reserves, low production cost and high specific capacity make these materials fit the current development demands of high-endurance power batteries. Focusing on the application of lithium-rich manganese-based cathode materials in lithium-ion batteries, this paper systematically summarizes the strengths and weaknesses of materials with four types of morphological features and their influences on battery electrochemical performance. By sorting out and comparing performance differences with commercial cathodes including traditional layered oxides, spinel structures and polyanionic materials, this paper aims to illustrate the development potential of lithium-rich manganese-based cathode materials in the field of high-specific-energy batteries. In summary, lithium-rich manganese-based materials are among the highly competitive cathode materials for next-generation lithium batteries. By organizing existing preparation processes and performance studies, this paper clarifies the correlations among preparation techniques, micro-morphology and electrochemical performance. It also provides theoretical foundations and optimization ideas for the subsequent development of novel cathode materials with high stability and durability via doping, surface coating and composite modification.

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