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Developing low-cost rechargeable batteries: beyond traditional layered oxide cathodes for Li-ion and beyond Li-ion batteries

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Abstract

The rising demand for energy storage systems, driven by the rapid adoption of electric vehicles and the global shift toward renewable energy, necessitates continuous efforts to lower the cost of current lithium-ion batteries (LIBs) and enhance the sustainability of existing battery chemistries. This feature article examines the key challenges associated with Ni- and Co-containing LIB cathodes and compares advancements in cathode development for non-traditional Li-ion and beyond Li-ion chemistries. First, a review of earth-abundant element-containing disordered rock-salt cathodes is presented, with a discussion of key strategies such as compositional tuning and carbon coating to improve their electrochemical performance. Hurdles in developing oxide-based cathodes for Na- and K-ion batteries are also highlighted, followed by an in-depth overview of polyanion and Prussian blue cathodes for Na- and K-ion systems. Overall, this article provides a systematic perspective on the design of earth-abundant, low-cost, and sustainable cathode materials for both LIB and beyond LIB technologies.

1. Introduction

The global shift toward electrification and decarbonization is driving unprecedented demand for efficient and sustainable energy storage solutions. The widespread adoption of portable electronic devices, the rapid growth of electric vehicles, and the increasing integration of intermittent renewable energy sources such as solar and wind into modern power grids are placing ever greater demands on rechargeable battery technologies. In response to this surging demand, the development of low-cost, high-performance rechargeable batteries has garnered significant attention worldwide. Although lithium-ion batteries (LIBs) have revolutionized the energy-storage landscape over the past few decades, there is now a more urgent need than ever to go beyond incremental improvements and explore new chemistries that offer advantages in terms of sustainability, cost, and scalability.¹⁻⁴ In addition to these, enhancing energy density remains a crucial performance target for enabling the widespread adoption of the technology. Beyond its direct impact on battery size, higher energy density can indirectly advance cost-effectiveness and sustainability objectives by reducing raw material requirements per unit of stored energy (related to \$ per kWh) and lowering associated environmental burdens throughout the product lifecycle.

LIBs based on layered transition-metal (TM) oxide cathodes, such as LiCoO_2 , LiNiO_2 , and their mixed metal derivatives (including $\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$ (NMC) and $\text{LiNi}_x\text{Co}_y\text{Al}_z\text{O}_2$ (NCA)), have underpinned the commercial success of this technology because of their high specific energy (Wh kg^{-1}) and long cycle life.⁵⁻⁹ However, these materials are increasingly viewed as unsustainable because they heavily rely on cobalt (Co) and nickel (Ni), two critical elements. As shown in Figure 1a, Co and Ni, along with Li, are neither earth abundant nor low cost.¹⁰⁻¹³ In particular, Co is

associated with volatile pricing, limited global reserves concentrated in geopolitically unstable regions, and problematic mining practices that raise serious concerns over human rights and environmental degradation.¹⁴⁻¹⁶ Ni, although more abundant, also faces growing supply constraints as demand intensifies for high-Ni cathodes in high-energy applications.^{17, 18} These issues present barriers to the large-scale deployment of LIBs, especially in emerging markets and grid-level energy storage where cost sensitivity is paramount. Therefore, the development of rechargeable batteries consisting of earth-abundant and low-cost elements, such as manganese (Mn) and iron (Fe) as redox centers, as well as sodium (Na) and potassium (K) as intercalating ions, has gained momentum as a promising avenue for achieving more sustainable and affordable energy storage.

The production capability of essential LIB components is another important parameter. The annual production of Co and Ni is ~0.3 million and ~3.7 million metric tons, respectively (Figure 1b).^{13, 19,}

²⁰ Assuming a single TM LiMO_2 (M = transition metal) with $\sim 800 \text{ Wh kg}^{-1}$, these annual production amounts can supply ~ 0.4 and $\sim 4.9 \text{ TWh/year}$ of LIB production, respectively. Given the 2024 LIB manufacturing capacity ($\sim 3 \text{ TWh/year}$),²¹ Co is not an option, and the Ni supply is also at risk. In addition, the estimated worldwide LIB manufacturing demand in 2030 is $\sim 6.8 \text{ TWh}$.²⁰ Neither Co nor Ni production can meet this surging demand. In sharp contrast, the annual production of a few TMs, such as Fe, Mn, titanium (Ti), and chromium (Cr), surpasses the future LIB manufacturing capacity.^{13, 19} For example, the annual production of Fe ($\sim 2,500$ million metric tons) could supply approximately $3,400 \text{ TWh}$, and that of Mn (~ 20 million metric tons) could supply $\sim 27 \text{ TWh}$. Therefore, it is likely that the price of Ni and Co will remain high as the battery industry grows, whereas the price of Mn and Fe would not be significantly affected by battery

production. This analysis clearly indicates that the development of Co- and Ni-free cathodes is essential, and no longer optional, for the battery industry.

This feature article will review recent efforts in developing Co- and Ni-free cathode materials for rechargeable batteries, focusing on (i) Mn-based disordered rock-salt oxides and oxyfluorides and (ii) beyond LIB chemistries, including Na- and K-ion chemistries. The status, important challenges, recent progress, and outlook will be discussed.

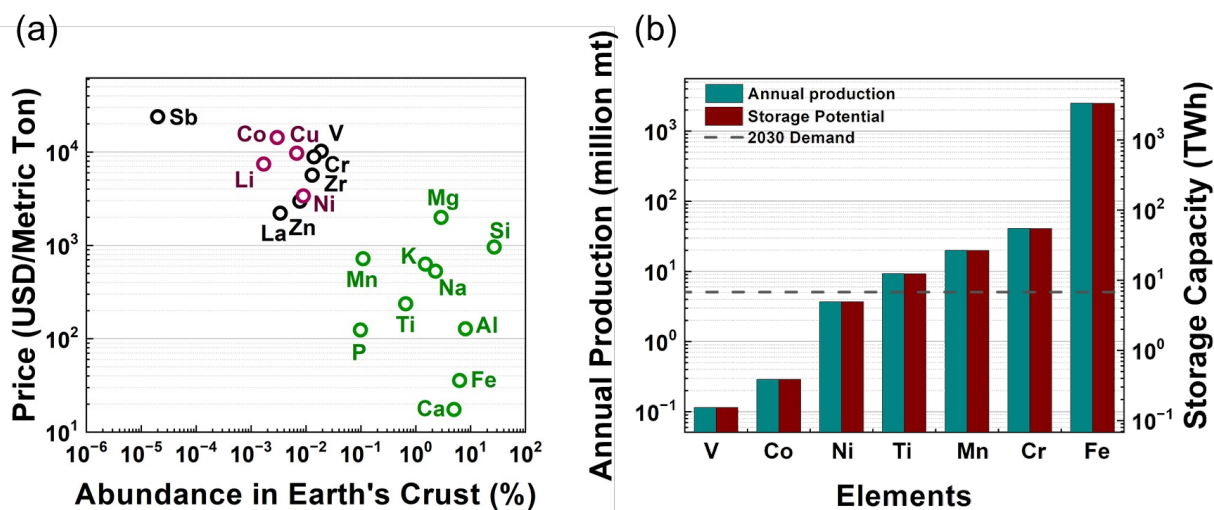


Figure 1. (a) Price and abundance of elements.^{10, 11, 13} Data was accessed on June 10th, 2025. (b) Annual production and storage capacity of TMs^{13, 19, 20} Data was accessed on June 10th, 2025. Storage capacity was estimated by assuming a single TM LiMO₂ (M = transition metals) with ~800 Wh kg⁻¹.

2. Disordered rock-salt oxides and oxyfluorides for LIBs

Layered oxide compounds, such as LiCoO₂, NMC, and NCA, are the traditional cathode active materials (CAMs) for LIBs. These layered oxide compounds crystallize in an α -NaFeO₂-type

layered structure (R-3m symmetry), where Li and TM cations occupy alternating layers in an ordered fashion in a close-packed oxygen framework (Figure 2a).²²⁻²⁵ This structural ordering enables fast, two-dimensional lithium diffusion and provides a stable framework during Li-ion intercalation and deintercalation, contributing to high energy density and good cycling stability.²⁶⁻²⁸ Despite their success, layered oxide compounds possess several critical drawbacks, including the dependence on expensive and supply-constrained elements such as Co and Ni and limited flexibility in accommodating alternative, more abundant TMs, such as Mn and Fe.

As a potential solution to overcome these limitations, the disordered rock salt (so-called DRX) cathode class has recently gained substantial attention since DRX compounds were first introduced as viable cathode materials for LIBs in the early 2010s by Ceder and colleagues.²³ DRX materials share a structural backbone with layered oxides in which both the anion and cation are based on close-packed oxygen frameworks and octahedral coordination of cations. However, the key difference lies in the cation arrangement. In DRX compounds, Li and TM ions are randomly distributed over the cation sites of a rock-salt-type structure, as shown in Figure 2b, rather than being segregated into distinct layers. Importantly, DRX compounds have more flexibility in accommodating TM species than layered oxide compounds. Whereas Co and Ni are required to stabilize the ordered layered oxide structure, DRX compounds can be stabilized without these expensive elements,²⁹⁻³¹ which unlocks the potential for earth-abundant and low-cost LIB cathode development.

A critical breakthrough in the development of DRX cathodes was the recognition that a lithium-excess composition is essential to enable percolating Li-ion diffusion pathways in the absence of long-range cation ordering. In both layered and DRX structures, Li ions migrate between

neighboring octahedral sites via an intermediate tetrahedral site, following an octahedral–tetrahedral–octahedral (o–t–o) pathway.^{23, 32–34} The migration barrier along this path is strongly influenced by the number of TMs that face share with the intermediate tetrahedral site.^{23, 35, 36} In ordered layered oxides, favorable 1-TM channels, where the tetrahedral site is coordinated by one TM and three Li ions, naturally form a two-dimensional percolating network for Li diffusion.³⁵ In contrast, DRX materials lack this long-range cation ordering and thus rely on a three-dimensional network of locally favorable diffusion channels. Specifically, 0-TM and 1-TM channels, where the tetrahedral site is face shared by zero or one TM, are essential for Li-ion diffusion.³⁵ Higher TM-coordinated pathways (such as 2-TM, 3-TM, and 4-TM channels) exhibit prohibitively high migration barriers due to strong electrostatic repulsion between migrating Li ions and face-sharing TM cations, making them effectively inaccessible.^{37–39} To ensure the formation of a continuous network of 0-TM and 1-TM pathways, a lithium-excess composition is required in DRX compounds. This requirement has been confirmed through both computational modeling and experimental validation,^{27, 40, 41} and it has since become a foundational design principle for DRX cathodes. Rather than relying on structural order, modern DRX design now focuses on compositional tuning and local short-range order (SRO) to achieve efficient Li-ion transport. This paradigm shift has enabled the development of a wide variety of Co- and Ni-free DRX cathodes with high capacities and compositional versatility, making DRX a compelling platform for next-generation sustainable energy storage.

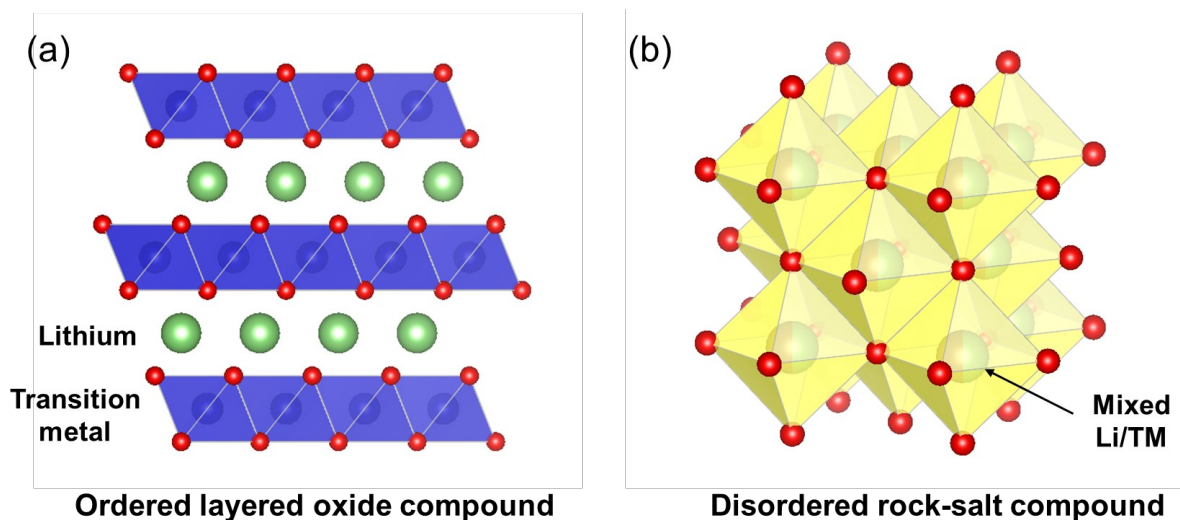


Figure 2. Crystal structures of (a) ordered layered oxide compound and (b) DRX compound.

2.1. Compositional tuning of Mn-based DRX cathodes

Although DRX cathodes can incorporate a wide range of redox-active TMs, including Fe, V, Cr, Ni, Mo, and Mn,^{22, 24, 42-47} this discussion focuses on Mn-based DRX materials. Mn is by far the most widely studied and utilized redox-active element in DRX cathodes owing to its natural abundance, low cost, and the possibility of accessing a two-electron redox process spanning Mn^{2+} to Mn^{4+} . These characteristics make Mn-based DRX compounds particularly attractive for high-capacity, resource-sustainable LIBs.

Mn-based DRX cathodes offer the potential for high energy density but often suffer from limited cycling stability and rate capability.^{26, 37, 48-51} These performance limitations are largely attributed to irreversible oxygen redox activity, sluggish Li-ion transport, and structural degradation under electrochemical cycling.⁵²⁻⁵⁷ To overcome these challenges, compositional tuning has emerged as a critical strategy for improving both the reversible capacity and cycling stability.

2.1.1. Lithium excess

As discussed above, lithium excess is a fundamental requirement in DRX systems to enable a percolating three-dimensional Li-ion transport network composed of 0-TM and 1-TM channels.^{23, 32, 36} These low-barrier diffusion pathways are essential for maintaining acceptable rate performance in the absence of long-range cation ordering. Therefore, in general, higher Li excess tends to improve the rate capability. However, when the Li-excess level increases in the DRX, the oxidation state of Mn increases proportionally for a fixed Mn content, decreasing the Mn redox capacities (Figure 3a). The Mn redox reaction is more reversible than the oxygen redox reaction, which often triggers irreversible oxygen gas evolution and permanent structure degradation.^{28, 58} Therefore, higher Li excess can reduce cycle life unless the compound is highly fluorinated to decrease the valence state of Mn, which will be further discussed in a later section.

As discussed above, there is an important trade-off regarding the Li-excess level in DRX compounds. Although lower Li-excess compositions can negatively impact the Li diffusion percolating within the DRX structure, they can also contribute to improved cycling performance. One potential strategy is to develop small-particle DRXs (a few hundred nanometers, similar to LiFePO_4) to reduce the lithium diffusion length while maintaining lower Li-excess compositions as proposed by Lee *et al.*⁴⁰ However, nano-sized DRX compounds are typically synthesized using a high-energy mechanochemical approach or at least the particle size is reduced by high-energy ball milling. Both processes can create substantial amounts of surface defects, accelerating unwanted side reactions in the electrochemical cells. Therefore, alternative synthetic routes should be considered to control the particle size of DRX compounds without damaging them. Sol-gel, combustion, and molten-salt synthesis approaches could be potential solutions.^{43, 57, 59} Although it is

less important for low-voltage TM DRX compounds (such as Mn-based DRX), Lee *et al.* pointed out that a Li-excess composition is necessary to make Li extraction more accessible at reasonable voltages (4.3–4.6 V) from high-voltage TM DRX compounds such as Fe^{3+} , Co^{3+} , and Ni^{3+} .⁴⁰

2.1.2. Mn content

The Mn content does not significantly affect the theoretically accessible Mn-redox capacities at a fixed Li-excess level (Figure 3b). Higher Mn content seems to result in higher Mn-redox capacities at first glance. However, the average oxidation state of Mn in the DRX structure increases as the Mn content increases. Therefore, the theoretically accessible Mn-redox capacities remain the same even though the redox-active Mn component increases in the DRX structure. However, one should not ignore another intriguing role of Mn-rich compositions. Mn-rich DRX compounds, which typically have ≥ 0.6 Mn per formula unit of DRX, transform into a partially disordered spinel-like structure (the so-called delta phase) upon repeated electrochemical cycling.^{31, 49, 60} This delta transformation is accompanied by substantial changes in the voltage profiles: (i) an increase in the overall capacity and (ii) the evolution of distinct plateaus at ~ 3 and ~ 4 V, as shown in Figure 3c.⁴⁹ This delta transformation often improves the Li diffusion kinetics, as the spinel-like cation configurations form more 0-TM sites, which are preferred for Li percolation compared to the random distribution in the DRX structure.

2.1.3. Redox-inactive metal species

Intriguingly, a redox-inactive metal species, which has a higher-valent d0 metal, also seems to play a pivotal role in the delta transformation from the DRX structure. Figure 3d summarizes Mn-based DRX compounds with varied Li and Mn contents and shows whether they are transformed into the delta phase after repeated electrochemical cycling, depending on their compositions.^{31, 40, 43, 45, 49, 59-65}

In this analysis, we determined that the delta transformation occurs when a plateau at ~4 V is clearly observed in the charge–discharge profiles. When Ti^{4+} is used as the high-valent d0 metal, the transformation from the DRX to the delta phase can occur for a Mn content > 0.5 . In contrast, the Mn content must be higher than 0.7 for the delta transformation to occur when Nb^{5+} is used. Although more in-depth studies are required, we speculate that Ti^{4+} can migrate into the Li sites in the DRX structure, similar to the case of layered oxides,⁶⁶⁻⁶⁸ which contributes to the delta transformation. In contrast, Nb^{5+} might not be prone to migrate into the Li sites because of its larger ionic size and higher valence states than Ti^{4+} .

Redox-inactive metals also play a critical role in determining lithium diffusion in DRX compounds by controlling the SRO. Ji and her colleagues directly compared two DRX compounds, $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Zr}_{0.4}\text{O}_2$ (LMZO) and $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_2$ (LMTO), and found that the Li percolating network was significantly affected by the d0 metal species.²⁷ Experimentally, they proved that the LMTO showed much higher Li diffusivity than LMZO, which is attributed to their different SRO. In addition, structure characterization and computational analysis demonstrated that LMTO contains more 0-TM channels than LMZO, and these channels are vital for Li diffusion. Recently, Song *et al.* argued that a lower content of d0 metal (Ti^{4+}) resulted in a lower Li-ion diffusion barrier based on experimental observation and computational analysis.⁶⁵

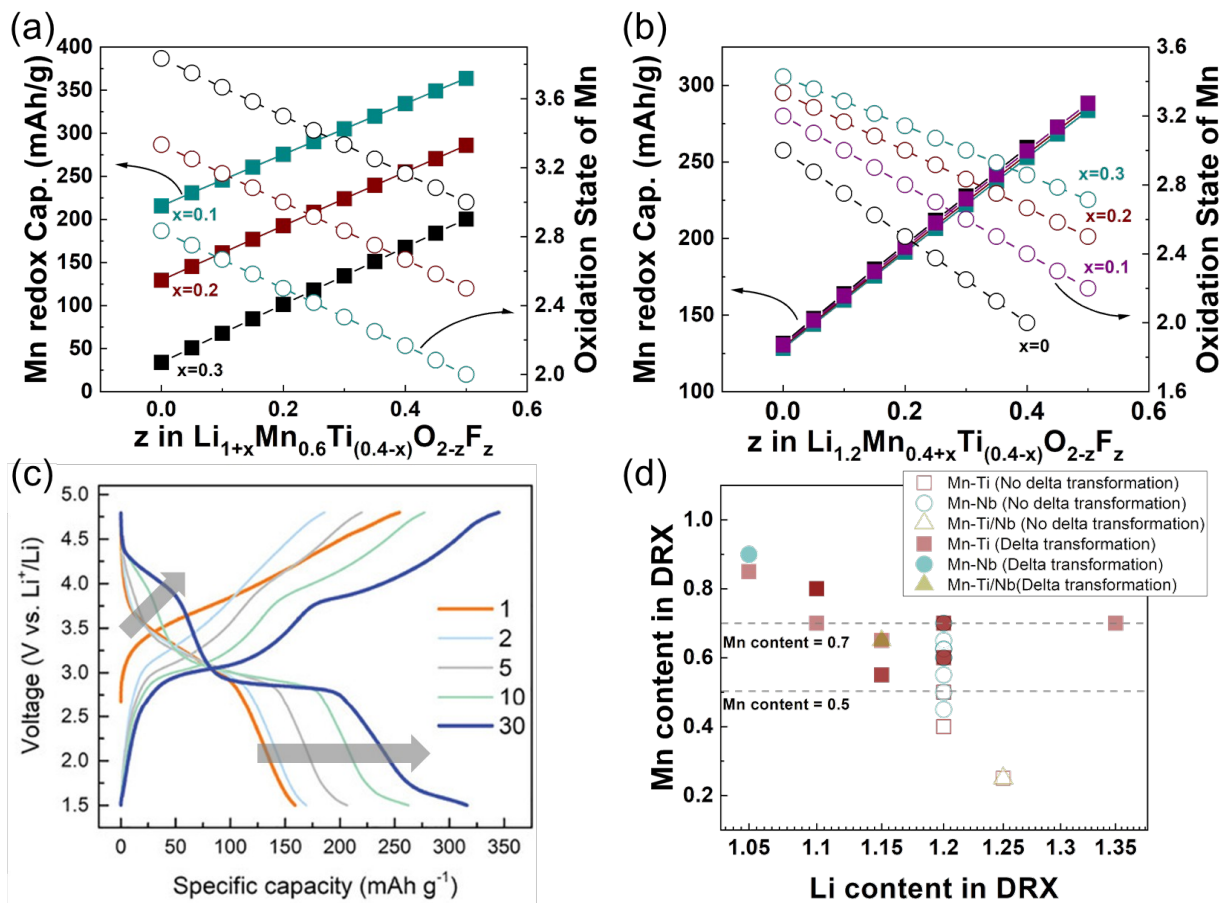


Figure 3. Oxidation state of Mn ions and corresponding cationic redox (theoretical) capacity as a function of F content in (a) $\text{Li}_{1+x}\text{Mn}_{0.6}\text{Ti}_{(0.4-x)}\text{O}_{2-z}\text{F}_z$ and (b) $\text{Li}_{1.2}\text{Mn}_{0.4+x}\text{Ti}_{(0.4-x)}\text{O}_{2-z}\text{F}_z$ DRX compounds. (c) Charge and discharge voltage profiles of $\text{Li}_{1.1}\text{Mn}_{0.8}\text{Ti}_{0.1}\text{O}_{1.9}\text{F}_{0.1}$ cathode (CAM:acetylene black:PVDF = 7:2:1) for the 1st (orange), 2nd (light blue), 5th (grey), 10th (green), and 30th (blue) cycles operated in the voltage range of 1.5–4.8 V at a current density of 10 mA g^{-1} ⁴⁹ Reproduced from ref. 49 with permission from Wiley ref. 49 Copyright 2023 . (d) Li and Mn contents that enable the delta transformation in various Mn-based DRX compounds. ^{31, 40, 43, 45, 49, 59-65} The open symbols indicate that the DRX does not transform into the delta phase after cycling, whereas the closed symbols indicate that the DRX does transform.

2.1.4. Fluorination

DRX compounds uniquely allow fluorination, whereas the fluorine substitution into traditional layered oxide compounds is thermodynamically unfavorable.^{29, 69} In general, the substitution of oxygen with fluorine in the layered oxide bulk does not occur, with the formation of F-rich (such as LiF) coating layers occurring instead.⁶⁹ In contrast, fluorination into the DRX bulk is feasible because Li excess and cation disorder create oxygen sites that are coordinated with Li-rich environments.²⁹ These oxygen sites are preferable for fluorine substitution. Importantly, fluorination has proven effective in improving the electrochemical properties of DRX compounds.^{26, 46, 58, 70} For example, the substitution of oxygen with fluorine reduces the valence state of redox-active Mn, which enables higher Mn-redox capacity and reduces detrimental oxygen oxidation and oxygen-release reactions (Figures 3a–b).²⁶ More comprehensive effects of fluorine substitution in DRX compounds have been well documented in a recent review article.²⁹ In this respect, many groups have studied and synthesized fluorinated Mn-based DRX compounds.^{26, 37, 50, 54, 58, 61, 62, 64, 70–74} Early studies used mechanochemical routes to synthesize fluorinated Mn-based DRX cathode compounds, which could substitute fluorine into the DRX structure up to ~34%^{26, 37, 50, 64} Such high fluorination levels are indeed far beyond the earlier computationally predicted fluorine solubility in DRX (up to 15%)⁷⁵, which indicates that the highly fluorinated DRX compounds are likely thermodynamically metastable. Although the fluorination is beneficial for improving the stability of the DRX cathode compounds and the mechanochemical synthesis process is effective in incorporating fluorine into the DRX bulk, it is worth noting that several challenges remain, including the scalability and reproducibility of the mechanochemical synthesis. Therefore, a solid-state synthesis process has been frequently used to synthesize fluorinated DRX compounds.^{58, 61, 71.}

^{72, 75} However, recent studies by Wu *et al.*, Giovine *et al.*, and Koirala *et al.* claimed that quantification of the fluorine incorporation into the DRX structure must be carefully performed when solid-state synthesis is applied because fluorine tends to evaporate and form low-crystalline (or amorphous) LiF at high temperature above 900 °C, which is required for DRX phase formation.^{63, 76, 77} In general, the solid-state synthesis produces significantly lower fluorine incorporation than the targeted compositions. Szymanski *et al.* attributed the difficulty in incorporating fluorine into the DRX structure to the following reasons (Figure 4a):⁷⁸ (i) LiF, which is a typical fluorine source in DRX synthesis, is volatile at > 848 °C. However, the formation of Mn-based DRX compounds in most cases completes above 900 °C. Therefore, the fluorine loss during the high-temperature synthesis process is unavoidable. (ii) Although different fluorine sources, such as MnF₂, are used to maintain a high fluorine chemical potential and improve fluorine solubility, LiF formation is difficult to avoid because fluorine sources react with the Li-containing precursor to form LiF at moderate temperatures below 400 °C. (iii) Mn–F bonds are thermodynamically less favorable than Li–F bonds. Therefore, when Mn is incorporated into the DRX formation, the fluorine tends to form LiF, limiting fluorine solubility in Mn-based DRX compounds.

In a recent study by Avvaru *et al.*, the fluorination level in Mn-based DRX compounds was significantly increased, achieving up to > 0.3 F⁻ per formula unit (>15%), which is more than twice as high as the level reported in previous solid-state synthesis.⁷⁰ When traditional precursors, such as Li₂O, LiF, MnO, and TiO₂, were used, *in situ* X-ray diffraction analysis identified the formation of intermediate phases, including Li₂(Mn,Ti)O₃, LiMnO₂, and Mn₃O₄, which tend to trap Mn in oxygen-rich environments, limiting the incorporation of fluorine (Figure 4b). In addition, these intermediate phases persist until the synthesis temperature reaches above that required for fluorine

volatility, which is the primary source of fluorine loss. In contrast, Avvaru *et al.* suppressed these intermediates and stabilized the fluorinated Mn-based DRX phase at a relatively low synthesis temperature (800 °C), thereby minimizing fluorine volatilization and maximizing fluorine incorporation (Figure 4c) by utilizing an unconventional precursor set, Li_6MnO_4 , MnF_2 , and TiO_2 .⁷⁰ Figure 4d summarizes the gap between targeted and quantified fluorine levels in Mn-based DRX compounds synthesized by solid-state process or microwave-assisted solid-state reaction.^{43, 63, 70, 76, 77} In this analysis, we only considered previous reports that quantified the fluorination levels in Mn-based DRX compounds. Although various fluorine concentrations (5—15 at%) were considered in the Mn-based DRX compound synthesis, the actual (quantified) fluorine contents in the DRX bulk were much lower than the targeted compositions. The highest quantified fluorine content in the DRX bulk was 6.5 at% in the literature.⁷⁷ Such a relatively high fluorine incorporation was only achieved for a low-Mn composition ($\text{Li}_{1.25}\text{Mn}_{0.20}\text{Ti}_{0.55}\text{O}_{1.85}\text{F}_{0.15}$). Interestingly, low-Mn DRX compounds showed higher quantified fluorination levels and smaller discrepancies from the targeted fluorine levels. This might be related to the fact that Mn–F bonds are thermodynamically less favorable than Li–F bonds. For low-Mn compositions, a smaller number of less favorable Mn–F bonds in the DRX structure are expected at a fixed fluorination level. Another factor that should be considered is that low-Mn DRX requires a lower synthesis temperature, which might reduce the fluorine evaporation risk.⁷⁷ Importantly, the alternative solid-state synthesis route proposed by Avvaru *et al.* successfully incorporates substantially higher fluorine ions into the DRX bulk (up to 17 at%),⁷⁰ which is well beyond the fluorination limit reported in previous literature. This finding signifies the importance of precursor selection and synthesis-process modification in determining reaction pathways, thereby controlling the fluorine solubility in the DRX bulk.

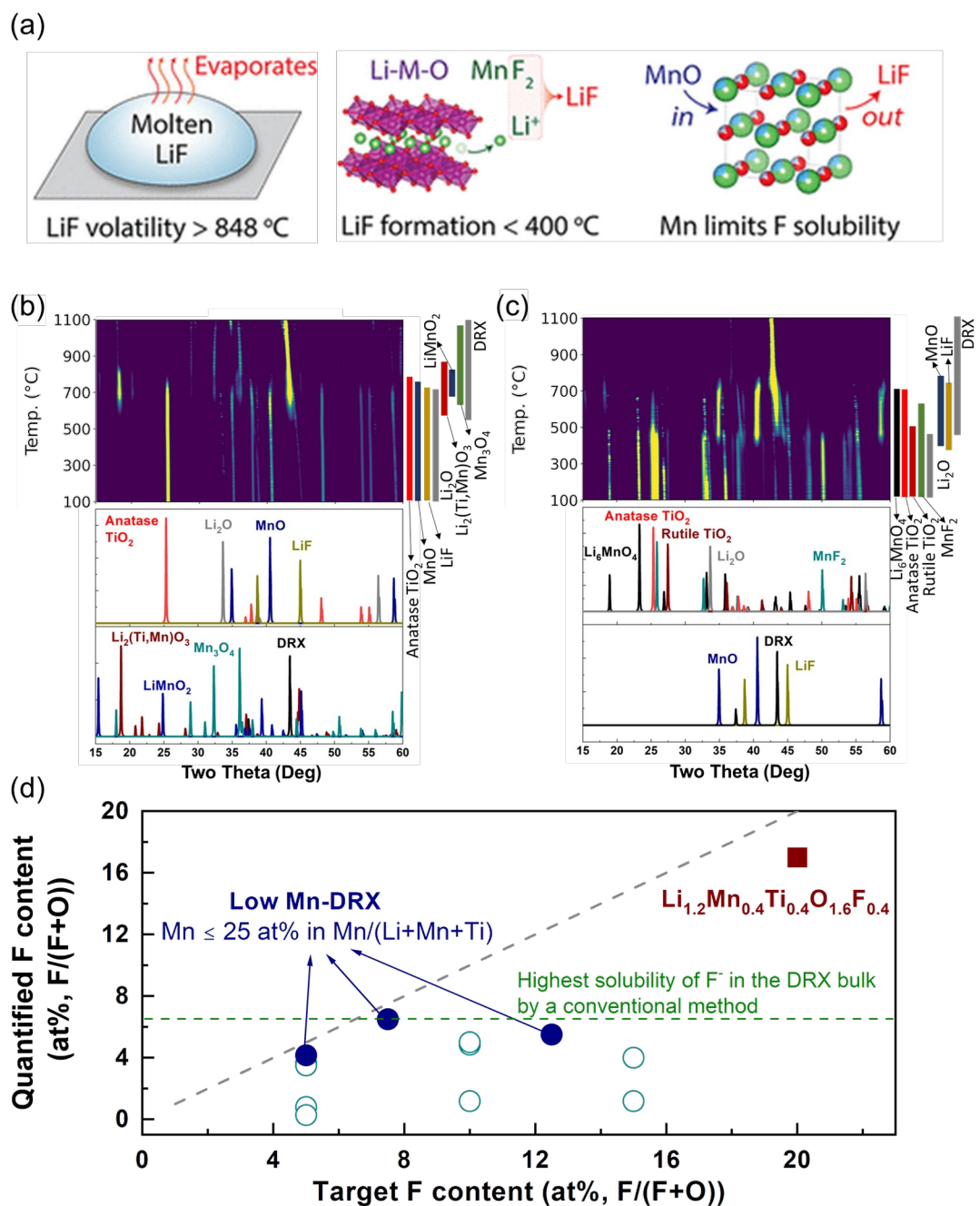


Figure 4. (a) Illustration showing the challenges of fluorination in DRX: formation of LiF below 400 °C, LiF release due to MnO incorporation into the DRX structure, and LiF evaporation ⁷⁸

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. *In situ* synchrotron thermal XRD measurements (converted to Cu K for better readability) captured during the synthesis of $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_{1.6}\text{F}_{0.4}$ (LMTOF1244) using precursors of (b) Li_2O , LiF , MnO , and TiO_2 and (c) Li_6MnO_4 , MnF_2 , and TiO_2 .⁷⁰ Reproduced from ref. 70 with permission from Wiley ref. 70 Copyright 2025 . (d) Gap between targeted and quantified fluorine levels in Mn-based DRX compounds synthesized via conventional methods (blue) and an alternative route (brown)^{43, 63, 70, 76, 77}

2.1.5. Short-range cation ordering (SRO) and its influences:

Short-range cation order (SRO) critically governs Li-ion transport kinetics and electrochemical performance in cation-disordered rocksalt cathodes. Although DRX lattices are globally disordered, local cation correlations generate non-random environments that modulate Li percolation through 0-TM (transition-metal free) channels. In an ideal random DRX lattice, ~9 % Li excess is required for percolation; however, SRO can either facilitate or obstruct Li migration depending on its type and extent (Figure 5a–d).^{32, 79} Transition-metal chemistry strongly influences SRO formation where large or high-valent cations (e.g., Zr^{4+} , Nb^{5+}) favor tetrahedral-type SRO that impedes Li transport, while smaller cations promote Li segregation and reduce migration barriers.^{27, 54, 80} Recent atomic-scale computational investigations claimed that DRX structures exhibit three principal short-range structural configurations: tetrahedral, octahedral, and cubic, arising from distinct cation-cation correlations within the FCC sublattice (Figure 5b–d).⁷⁹ They argued that these configurations manifest as characteristic diffuse scattering features in reciprocal and diffraction space (Figure 5e–f), serving as fingerprints of local chemical environments. Among them, octahedral-type order promotes interconnected 0-TM Li pathways, enhancing Li diffusion and rate capability, whereas cubic-type SRO disrupts these percolation networks. In their

experimental validation, the fluorinated system ($\text{Li}_{1.2}\text{Ti}_{0.2}\text{Mn}_{0.6}\text{O}_{1.8}\text{F}_{0.2}$) exhibited a higher fraction of cubic order correlates with reduced Li mobility compared to the octahedral dominant $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_2$ phase,⁷⁹ illustrating the sensitivity of local ordering to both cationic and anionic substitution.⁸¹

Strategies such as rapid quenching, fluorination, and high-entropy cation mixing have proven effective in tuning SRO and improving Li kinetics.^{81, 82} In high-entropy DRX (HE-DRX) systems, the incorporation of multiple transition metals enhances configurational entropy, effectively minimizing the predominance of any single SRO patterns and promoting isotropic Li diffusion pathways. As shown in Figure 5g–i, increasing the number of transition-metal species from two (TM2) to six (TM6) progressively reduces the diffuse scattering intensity observed in electron diffraction, signifying a diminished degree of specific SRO and the emergence of a structurally homogeneous network conducive to efficient Li transport.⁸² Computational studies further indicate that site-energy disorder from random cation distributions modulates Li hopping barriers by orders of magnitude.^{83, 84} Collectively, these findings highlight that controlling SRO through compositional tuning and synthesis kinetics is pivotal to optimizing Li transport, redox reversibility, and cycling stability in DRX cathodes.

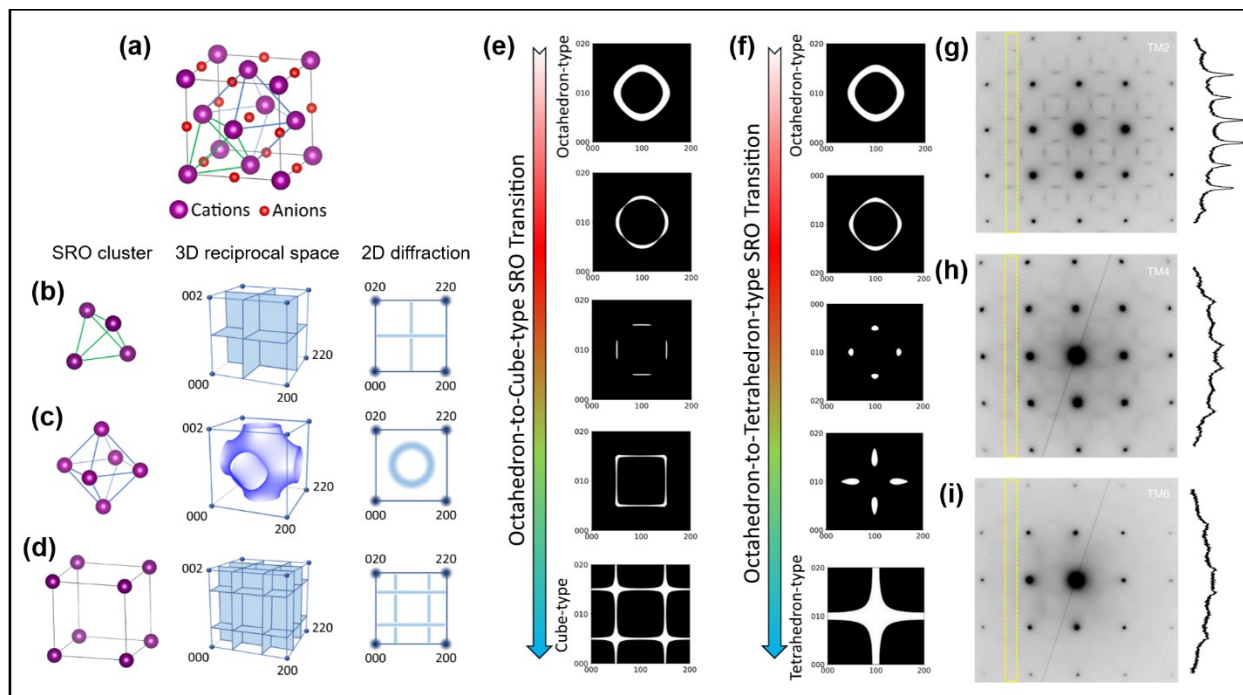


Figure 5. (a) Crystal structure of DRX, in which different atomic clusters are highlighted by the colored polyhedrons. Reproduced from ref. 79 with permission from Nature Communications ref. 79 Copyright 2023 (b) Octahedron-type SRO cluster, (c) cubic-type SRO cluster, and (d) tetrahedron-type SRO cluster (left), and the corresponding calculated diffraction locus in reciprocal space (middle) and diffraction patterns along [001] zone axis (right). Reproduced from ref. 79 with permission from Nature Communications ref. 79 Copyright 2023. Calculated diffraction locus for (e) the octahedral-to-cubic SRO transition and (f) octahedral-to-tetrahedral SRO transition. Reproduced from ref. 79 with permission from Nature Communications ref. 79 Copyright 2023. (g-i) TEM Electron Diffraction patterns collected on as-synthesized (g) $\text{Li}_{1.3}\text{Mn}^{3+}_{0.4}\text{Ti}_{0.3}\text{O}_{1.7}\text{F}_{0.3}$, (h) $\text{Li}_{1.3}\text{Mn}^{2+}_{0.2}\text{Mn}^{3+}_{0.2}\text{Ti}_{0.1}\text{Nb}_{0.2}\text{O}_{1.7}\text{F}_{0.3}$, (i) $\text{Li}_{1.3}\text{Mn}^{2+}_{0.1}\text{Co}^{2+}_{0.1}\text{Mn}^{3+}_{0.1}\text{Cr}^{3+}_{0.1}\text{Ti}_{0.1}\text{Nb}_{0.2}\text{O}_{1.7}\text{F}_{0.3}$. Reproduced from ref. 82 with permission from Nature Materials ref. 82 Copyright 2020.

2.1.6. Synthesis

DRX materials are typically synthesized via high-temperature solid-state routes (800-1100 °C), where thermal energy promotes cation disorder and phase stabilization. However, metastable compositions particularly those rich in fluorine or containing high-valent cations such as Mo⁶⁺, Nb⁵⁺, and V⁵⁺ often require non-equilibrium mechanochemical activation to induce disorder and expand the accessible compositional space.^{23, 47, 58, 85-87} Depending on the reaction pathway and thermodynamic environment, synthesis methods such as solid-state reactions^{25, 58, 70, 77, 79}, sol-gel processing⁴³, molten-salt^{60, 80, 88} mechanochemical^{26, 44, 47, 89}, and microwave-assisted^{63, 90} approaches yield varying degrees of crystallinity, SRO, and Li-diffusion. Each technique offers distinct advantages in particle-size control, compositional homogeneity, and scalability, yet presents challenges in optimizing cation disorder and electrochemical performance. The following section discusses these synthesis routes, highlighting their processing principles, advantages, and remaining challenges.

Solid-State Synthesis

Solid-state synthesis remains the most direct and scalable route to obtain DRX cathodes. Stoichiometric precursors are typically mixed and calcined at 900-1100 °C under an inert atmosphere to exceed the cation-disordering temperature, thereby stabilizing the DRX phase. This method yields highly crystalline materials with reproducible phase purity and compositional flexibility across systems such as Mn-Ti^{77, 79}, Mn-Nb^{58, 91}, and Mn-Ti-Cr⁹². However, the long diffusion paths inherent to bulk reactions often result in broad particle size distributions (10-25 µm) and limited control over SRO, requiring further post-treatment to enhance Li-ion transport.

Mechanochemical Synthesis

High-energy ball milling provides a versatile route to synthesize DRX cathodes by inducing cation mixing and defect formation through repeated mechanical impact and shear.^{26, 44, 47, 87} This technique enables the stabilization of metastable compositions and incorporation of high fluorine contents beyond the equilibrium solubility limit (e.g., $\text{Li}_{1+x}\text{TM}_{1-x}\text{O}_{2-y}\text{F}_y$). This mechanochemical process produces nanosized particles enriched with structural defects that facilitate Li-ion transport and enhance electronic conductivity, improving electrochemical performance. For instance, $\text{Li}_2\text{Mn}_{2/3}\text{Nb}_{1/3}\text{O}_2\text{F}$ synthesized by high-energy ball milling exhibits $\text{Mn}^{2+}/\text{Mn}^{4+}$ double-redox behavior, delivering exceptional capacity and energy density.²⁶ However, challenges such as uncontrolled surface reactivity, and increased electrolyte side reactions arising from the high surface area remain major bottlenecks for their large-scale adoption.

Molten-Salt Synthesis

Molten-salt synthesis represents a scalable approach for producing DRX materials with controlled morphology, uniform particle size, and high crystallinity.^{60, 80, 88} In this process, molten salts such as LiCl, KCl, NaCl, or KOH serve as ionic fluxes that enhance cation mobility and promote crystal growth within a liquid medium. Using such a molten-salt synthesis process, for example, Kan *et al.* successfully synthesized phase-pure and discrete $\text{Li}_{1.3}\text{Nb}_{0.3}\text{Mn}_{0.4}\text{O}_2$ single crystals with uniform morphology and size distribution.⁸⁸ However, a key limitation of this approach is its difficulty in producing sub-micron DRX particles, which are necessary to facilitate fast Li^+ diffusion. Optimizing salt composition, reaction temperature, and cooling kinetics could enable the synthesis

of submicron DRX powders, expanding the applicability of molten-salt synthesis to high-rate DRX cathodes.

Sol-Gel Synthesis

The sol-gel process enables atomic-level mixing of metal precursors, leading to homogeneous gels that decompose into a desired DRX phase upon calcination (typically 700-900 °C).⁴³ This molecular-scale uniformity enhances compositional control, reduces synthesis temperature, and yields fine particles with high phase purity. Compared to conventional solid-state routes, the sol-gel derived DRXs often exhibit improved cycling stability owing to their enhanced elemental homogeneity and reduced local strain. For example, Patil *et al.* synthesized $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_2$ at 900 °C and observed that DRX formation initiated at a moderate temperature (800 °C), with the sol-gel derived product showing superior cycling stability compared to solid-state.⁴³ Nonetheless, this process requires careful optimization of drying and annealing protocols for reproducible results and removing residual organics, which may impact the electrochemical properties.

Microwave-Assisted Synthesis

Microwave-assisted synthesis offers a rapid, and energy-efficient pathway for producing DRX materials, enabling phase formation within minutes.^{63, 90} This technique employs uniform volumetric heating to promote homogeneous nucleation and suppresses phase segregation. Recently, Wu *et al.* demonstrated the synthesis of $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_2$ and $\text{Li}_{1.3}\text{Mn}_{0.4}\text{Nb}_{0.3}\text{O}_{1-x}\text{F}_x$ using a microwave assisted solid state technique, achieving complete reaction in as short as 5 min.⁹⁰ Despite its efficiency and potential scalability, challenges such as limited temperature control and uneven electromagnetic field distribution can affect reproducibility. Continued advancements in reactor

design and process monitoring are expected to enhance heating uniformity and establish microwave-assisted synthesis as a viable large-scale route for DRX production.

Each synthesis strategy uniquely influences cation disorder, particle morphology, and the evolution of SRO, thereby governing Li-ion transport and redox dynamics in DRX cathodes. Solid-state and sol-gel methods emphasize scalability, phase purity, and compositional precision, while mechanochemical, molten-salt, and microwave-assisted techniques enable tunable defect chemistry and nanoscale architecture under controlled synthesis environments. Advancing DRX synthesis will require establishing quantitative correlations among synthesis processes, atomic-scale ordering, and electrochemical properties, facilitating the rational design of synthesis routes to achieve high-energy, high-rate, and structurally stable DRX cathodes.

2.2. Carbon coating and composite

DRX cathodes remain in an early stage of technological development, facing several processing challenges that limit their practical implementation. One of the most pressing issues is the need for a high carbon content, often in the range of 10–20 wt%, to compensate for their intrinsically low electronic conductivity.⁴⁸ This excess of carbon significantly reduces the active material fraction in the positive electrode, thereby lowering the electrode-level capacity and gravimetric energy density. Therefore, reducing the excessive carbon content in the coatings of DRX materials could significantly enhance their overall energy density. In addition, to achieve high electrode-level electrical connectivity, most studies have relied on high-energy ball milling to create homogeneous mixing of the composite.^{43, 57, 59, 90, 93-98} However, the high-energy ball-milling process may induce microstructural defects in DRX particles, including particle fragmentation, strain accumulation,

and surface disorder, which can degrade the long-term cycling performance. To overcome these challenges, the development of alternative strategies that can improve electronic percolation without excess carbon content and the creation of microstructural defects is needed.

Recent efforts have explored thin carbon coating/wrapping over DRX particles. Xu *et al.* applied amorphous carbon coating on $\text{Li}_{1.2}\text{Mn}_{0.4}\text{Ti}_{0.4}\text{O}_2$ particles *via* the vapor-phase method, which substantially increases the electrical conductivity.⁹⁵ However, a high-energy ball-milling process with carbon nanotubes (CNTs) was still required to deliver a reasonable capacity over 200 mAh g⁻¹. Zhou *et al.* developed carbon-coated $\text{Li}_{1.2}\text{Mn}_{0.2}\text{Ti}_{0.6}\text{O}_2$ *via* the sol–gel method with sucrose as a carbon source.⁹³ However, ball milling with excess carbon additive (20 wt%) was unavoidable during the electrode preparation for electrochemical testing. Recently, Patil *et al.* developed graphite-derived coatings, applied *via* high-energy ball milling, which ensures intimate contact with particle surfaces (Figure 6b).⁹⁴ Compared to other forms of carbon additives, the graphite-derived coating provided improved cycling stability (85.4% retention after 50 cycles with 10 wt% carbon), as shown in Figure 6a. In parallel, Avvaru *et al.* proposed a solution-based synthesis approach for graphene wrapping over $\text{Li}_{1.2}\text{Mn}_{0.6}\text{Ti}_{0.2}\text{O}_{1.8}\text{F}_{0.2}$ particles, where electrostatic attraction between negatively charged graphene sheets and positively charged polymer-coated DRX particles was used as the driving force to assemble the composite structure.⁵⁵ Although this approach produced uniform graphene wrapping over DRX particles (Figure 6d), the graphene-wrapped DRX cathode suffered from rapid capacity degradation (Figure 6c). The authors claimed that Li^+/H^+ exchange and TM dissolution occurred during the aqueous-based solution process, which damaged the DRX particles. Searching for alternative solvents and process optimization for conformal carbon coating/wrapping without damaging the DRX particles remains an important challenge. In

addition, the authors found that the DRX transforms into a spinel-like phase upon heating under an Ar environment with the presence of graphene. This finding may indicate the instability of DRX under a reducing environment and explain why conventional carbothermal reduction is not compatible with DRX cathodes.

Another interesting approach is the use of conductive additives with high geometric aspect ratios, such as multi-walled carbon nanotubes (MWCNTs). In a recent work, Lee and colleagues developed a DRX-MWCNT composite, where the high aspect ratio and mechanical resilience of the MWCNTs created robust electron percolating conductive networks that maintained electrical contact between the MWCNTs and DRX particles during cycling, even at low additive content (Figure 6e).⁹⁶ Notably, the MWCNT-containing DRX cathodes achieved high active material loadings exceeding 95 wt% and high specific energies $> 1000 \text{ Wh kg}^{-1}$. A few studies have also demonstrated the effectiveness of CNT-based DRX composites in improving the electron percolating networks of the positive electrodes.^{95, 99}

These advancements suggest that with careful tuning of the conductive architectures, through carbon coating or nanostructure engineering, it is possible to overcome key barriers in DRX electrode design and improve the electrode-level energy density. However, most DRX reports still use $>10 \text{ wt\%}$ carbon additive and require excessive shaker milling (or ball milling) to mix the DRX cathode active material and conductive carbon (Figure 6f).^{43, 55, 57, 59, 90, 93-98} To step closer to practical applications, further efforts are needed to eliminate the excessive milling process and reduce the carbon additive in the DRX electrode fabrication.

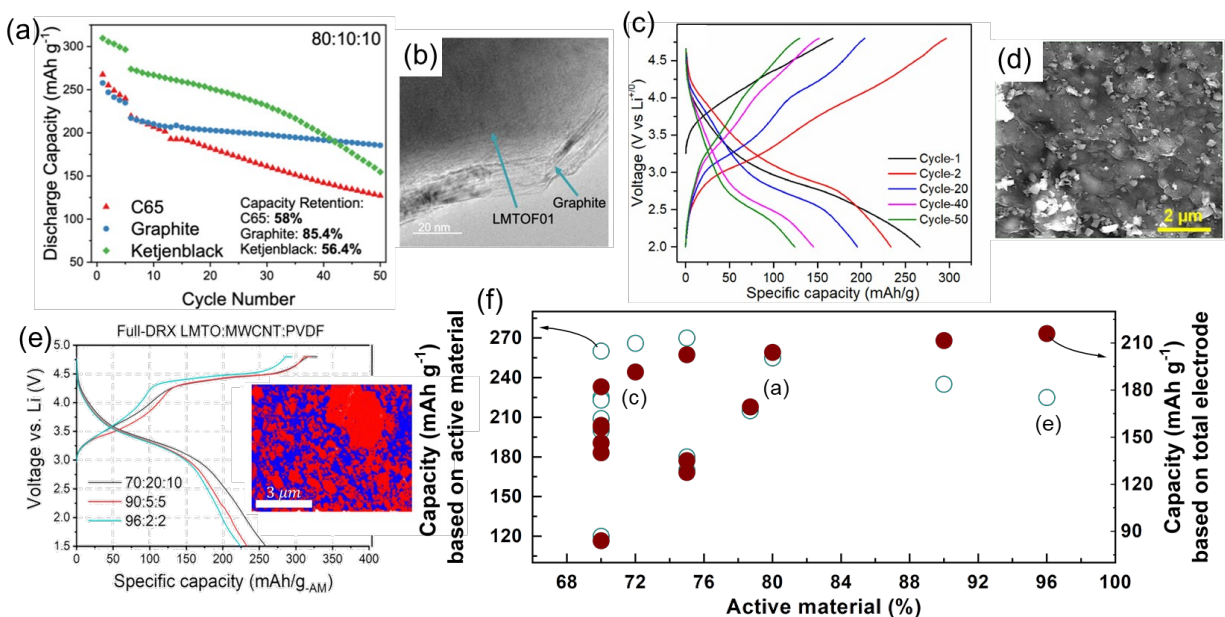


Figure 6. (a) Comparison of discharge capacity of Li_{1.2}Mn_{0.5}Ti_{0.3}O_{1.9}F_{0.1} cathodes with C65, graphite, and Ketjenblack conductive carbon (CAM:carbon:binder = 80:10:10) cycled between 1.5 and 4.8 V at a specific current of 10 mA g⁻¹ for the first five cycles and 20 mA g⁻¹ for the subsequent cycles.⁹⁴ Reproduced from ref. 94 with permission from American Chemical Society ref. 94 Copyright 2023. (b) TEM images of Li_{1.2}Mn_{0.5}Ti_{0.3}O_{1.9}F_{0.1} ball milled with graphite.⁹⁴ Reproduced from ref. 94 with permission from American Chemical Society ref. 94 Copyright 2023. (c) Galvanostatic charge-discharge profiles of 10 wt%-rGO-wrapped polyethyleneimine-coated Li_{1.2}Mn_{0.6}Ti_{0.2}O_{1.8}F_{0.2} cathode at current density of 20 mA/g.⁵⁵ Reproduced from ref. 55 with permission from Elsevier ref. 55 Copyright 2025. (d) SEM images of 10 wt%-rGO-wrapped polyethyleneimine-coated Li_{1.2}Mn_{0.6}Ti_{0.2}O_{1.8}F_{0.2}.⁵⁵ Reproduced from ref. 55 with permission from Elsevier ref. 55 Copyright 2025. (e) The first cycle voltage profiles of Li_{1.2}Mn_{0.4}Ti_{0.4}O₂ cathodes with varying MWCNT/PVDF content, cycled between 1.5 and 4.8 V at a specific current of 25 mA g⁻¹ with an inset of energy-dispersive X-ray spectroscopy (EDS) mapping of Li_{1.2}Mn_{0.4}Ti_{0.4}O₂ (red), MWCNT/PVDF (blue).⁹⁶ Reproduced from ref. 96 with permission from Royal Society of

Chemistry ref. 96 Copyright 2024 (f). Performance summary of Mn-based DRX cathodes in active material (wt%) in electrodes. [43](#), [55](#), [57](#), [59](#), [90](#), [93-98](#) Left y-axis: specific capacity per DRX mass (Blue, open circle). Right y-axis: specific capacity per total electrode mass (Brown, closed circle).

3. Cathode development for Na- and K-ion batteries

Given the resource constraints and price volatility of key components of LIBs, such as Ni, Co, and Li, alternative rechargeable batteries, including Na- and K-ion batteries, have attracted significant interest. Commercial LIB cathodes mainly utilize Co and Ni as the redox-active components. Although recent developments have moved toward higher Ni contents (50%–80%) to avoid Co and achieve higher energy density for LIBs, both key elements of Co and Ni drive up costs and raise supply chain concerns due to the reliance on critical materials^{1, 17, 100-103}. In contrast to Li-ion chemistries, the cathode materials for Na- and K-ion batteries can use earth-abundant elements (e.g. Mn, Fe, Mg, Cu, Zn, *etc.*), supporting more sustainable and scalable production of diverse cathode materials such as layered oxides, polyanionic frameworks, and Prussian blue analogues ¹⁰⁴⁻¹⁰⁶. In addition, Na-ion cathodes typically demonstrate superior thermal stability, although a more systematic evaluation in a full-cell system is still required, ¹⁰⁷ in contrast to high-Ni Li-NMC variants (e.g., NMC 622, NMC 811) that exhibit reduced thermal stability and are prone to oxygen release at elevated voltages, increasing the risk of thermal runaway. ^{108, 109}

The strategic advancement of these cathode materials presents new opportunities for developing cost-effective and sustainable battery technologies. In the following sections, we provide comparative evaluations of key parameters across available cathodes for Li-, Na-, and K-ion

systems, which can offer valuable insights into existing challenges and guide future efforts toward their optimization and improvement.

3.1. Limitations and strategies for traditional layered oxide-based cathodes for Na- and K-ion batteries

Layered oxide cathodes have attracted significant attention as cathodes for Na- and K-ion batteries [110-119](#) with the expectation that Na- and K-ion intercalation behaviors and properties would be similar to their Li counterparts, which deliver high energy density and excellent cycling performance. However, researchers face several intrinsic limitations of Na and K layered oxide cathodes. First, Li layered oxide compounds typically form a stoichiometric LiMO_2 (M = transition metals), stabilized in the O3-type structure. [35, 120-123](#) In sharp contrast, layered oxide cathodes for Na- and K-ion batteries typically crystallize in alkali-ion-deficient compositions: Na_xMO_2 and K_xMO_2 (M = transition metals, $x < 1.0$) in P2, O3, or P3 polymorphs. [119, 124-126](#) In K-ion systems, most layered compounds are inherently K deficient, which limits the amount of potassium available from the cathode for reversible de/intercalation. [127-131](#) To assemble rocking-chair-type full cells, a pre-potassiation process in either the cathode or anode is required. A rare stoichiometric O3-type KCrO_2 was stabilized by the strong octahedral-site preference of Cr^{3+} , which offset the destabilizing K^+-K^+ repulsion typical in fully potassiated structures. [128](#) In Na-ion systems, stoichiometric compositions are more common; however, many P2- and P3-type layered oxides form Na-deficient compositions similar to their K counterparts. [119, 123, 125, 130, 132, 133](#) Second, Na and K layered oxides present significantly sloped voltage profiles compared to their Li counterparts, as illustrated in Figure 7a. ¹ The sloped voltage curves limit the achievable specific capacity in a given voltage window, which lowers the specific energy and energy density of Na and K layered oxides compared with those of Li layered oxides. The sloped voltage profiles of Na and K layered oxides

originate from the stronger electrostatic Na^+-Na^+ or K^+-K^+ repulsion compared with that of Li^+-Li^+ in the layered oxide framework. More detailed explanations can be found in our previous review articles.^{1, 35} The strong electrostatic repulsion between mobile alkali ions influences the chemical potential of the alkali ions as a function of their concentration. The chemical potential change is referred to as the voltage in the electrochemical cell. When the chemical potential change is more significant with respect to the alkali ion concentration, a larger voltage swing is required to de/intercalate the same amount of alkali ions. Here, we quantify the average voltage swing required to de/intercalate 0.1 alkali ions, as shown in Figure 7b, following equation [1]:

$$\text{Voltage swing (for 0.1 alkali ion intercalation)} = \frac{[V_2 - V_1]}{[C_2 - C_1]} * 0.1 .$$

Equation [1]

Here, V_1 and V_2 are the lower and upper voltage limits, respectively, where the thermodynamic chemical potential change occurs with respect to the alkali ion concentration. Regions where a dramatic voltage change occurs due to the kinetic limitations are excluded from this analysis. C_1 and C_2 represent the smaller and higher alkali ion concentration limits, respectively, in which the thermodynamic chemical potential changes.

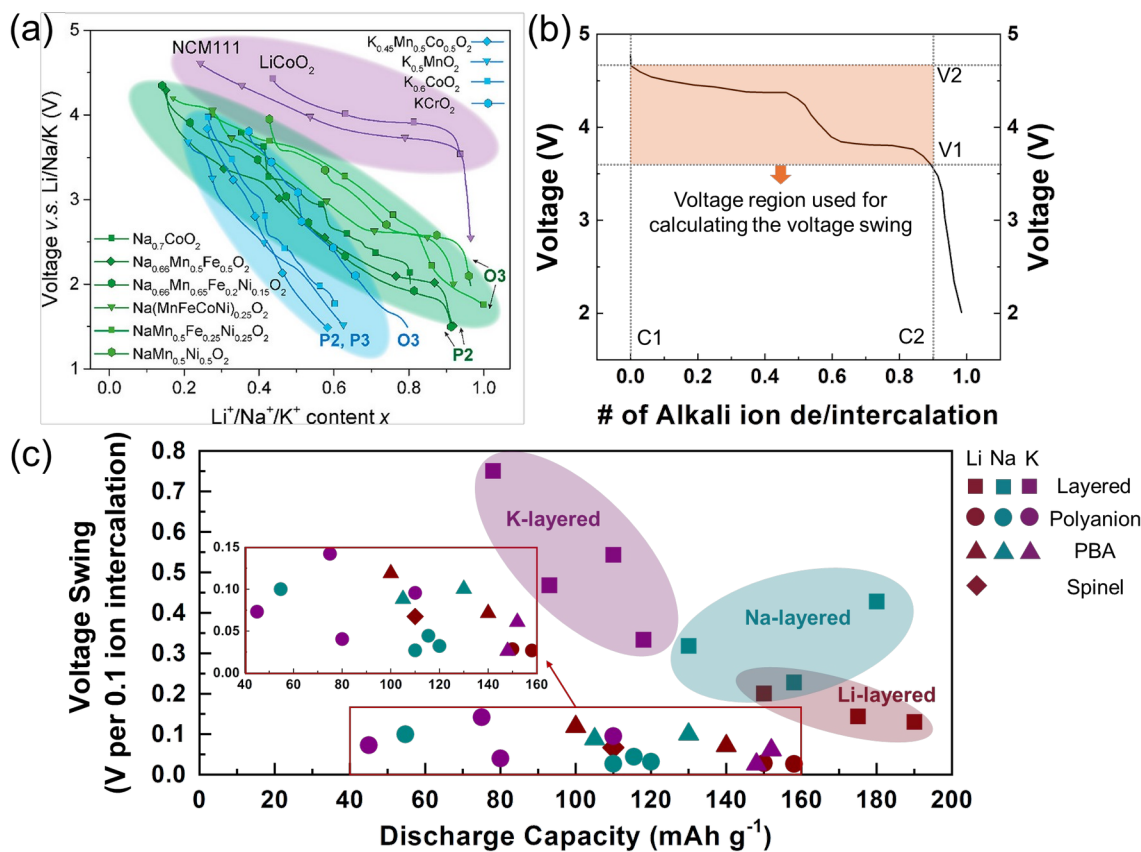


Figure 7. (a) Discharge profiles for various alkali-metal layered oxides. The shaded areas show that the voltage profiles have similar slopes for a particular alkali metal (Li, purple; Na, green; K, blue).¹ Reproduced from ref. 1 with permission from American Chemical Society ref. 1 Copyright 2021. (b) An arbitrary voltage curve as a function of alkali ion de/intercalation to exemplify how to calculate the average voltage swing to be used in (c). (c) Discharge capacity vs. average voltage swing plot of various cathode materials for Li-, Na-, and K-ion batteries.^{128, 134-157}

Figure 7c compares the voltage swing of layered oxides for Li-, Na-, and K-ion chemistries along with other types of cathodes such as polyanionic compounds and Prussian blue analogues (PBAs).^{128, 134-157} In contrast to Li layered oxides, Na- and K-ion layered oxides require much higher

voltage swings to de/intercalate 0.1 alkali ions. This finding indicates that the K layered oxide cathodes require a much wider voltage limit to de/intercalate the same amount of K^+ ions compared with cathodes based on Li and Na layered oxides. Because the electrochemical stability windows of both the cathode and electrolyte are limited, the voltage slope restricts the achievable specific capacity of the cathode. Whereas Na layered oxides have potential for performance improvement, K layered oxides have too low of a specific capacity to be utilized as cathodes due to the requirement for an extremely high voltage swing to extract a reasonable number of K^+ ions. The need for a higher cut-off voltage to access the high capacity of layered K oxides limits their cycling stability, primarily due to continuous electrolyte oxidation and concomitant structural deterioration.¹⁵⁸ In contrast, polyanionic compounds and PBAs, which have a larger open space for alkali ions, exhibit much smaller voltage swings. This difference can be attributed to the larger open space for alkali ions potentially reducing the electrostatic repulsion between the intercalated alkali ions.

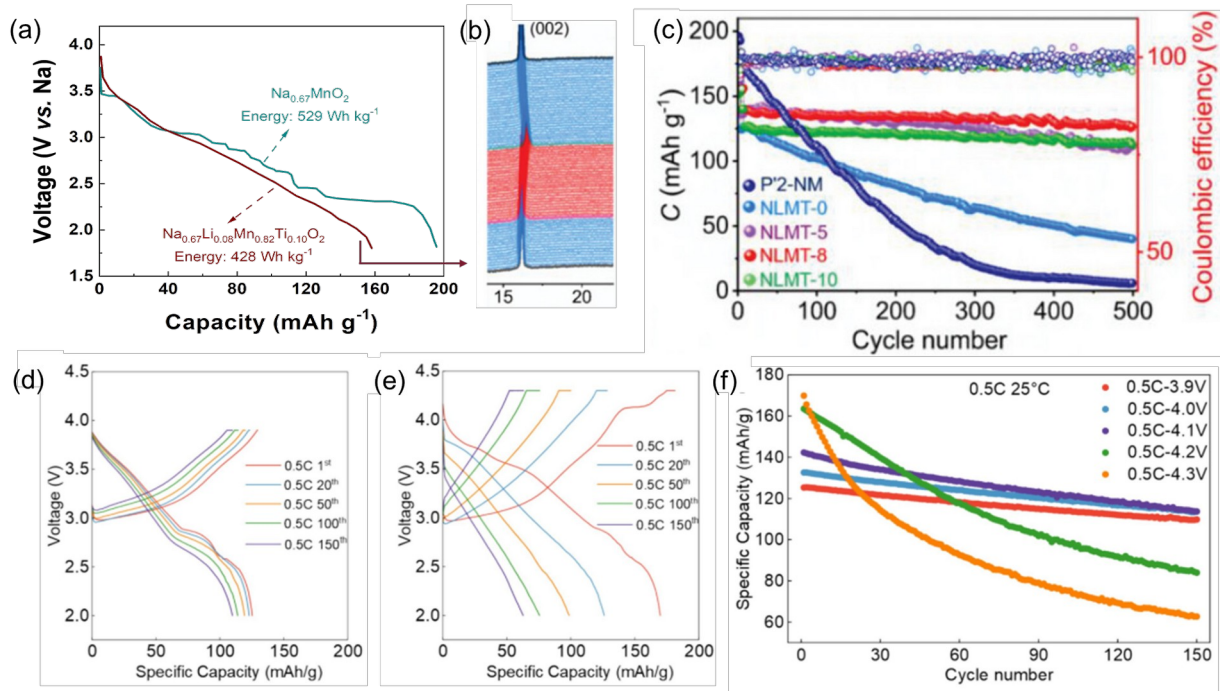


Figure 8. (a) Discharge curves of $\text{Na}_{0.67}\text{MnO}$ and $\text{Na}_{0.67}\text{Li}_{0.08}\text{Mn}_{0.82}\text{Ti}_{0.10}\text{O}_2$.¹⁵⁹ Reproduced from ref. 159 with permission from Wiley ref. 159 Copyright 2023. (b) *In situ* (002) peak evolution collected during the first charge/discharge and second charge of NLMT-8 at 20 mA g^{-1} between 1.8 and 4.3 V. Reproduced from ref. 159 with permission from Wiley ref. 159 Copyright 2023. (c) Cycling performance of $\text{Na}_{0.630}\text{Mn}_{0.908}\text{Ti}_{0.092}\text{O}_2$ (NLMT-0), $\text{Na}_{0.653}\text{Li}_{0.047}\text{Mn}_{0.858}\text{Ti}_{0.095}\text{O}_2$ (NLMT-5), $\text{Na}_{0.643}\text{Li}_{0.078}\text{Mn}_{0.827}\text{Ti}_{0.095}\text{O}_2$ (NLMT-8), $\text{Na}_{0.632}\text{Li}_{0.095}\text{Mn}_{0.806}\text{Ti}_{0.099}\text{O}_2$ (NLMT-10), and $\text{Na}_{0.649}\text{MnO}_2$ (P'2-NM)¹⁵⁹ Reproduced from ref. 159 with permission from Wiley ref. 159 Copyright 2023. Charge/discharge curves for the 1st, 20th, 50th, 100th, and 150th cycles at cutoff voltages of (d) 3.9 V and (e) 4.3 V for $\text{Na}/\text{NaNi}_{1/3}\text{Fe}_{1/3}\text{Mn}_{1/3}\text{O}_2$ cells.¹⁶⁰ Reproduced from ref. 160 with permission from Elsevier ref. 160 Copyright 2024. (f) Long-term cycling performance of $\text{Na}/\text{NaNi}_{1/3}\text{Fe}_{1/3}\text{Mn}_{1/3}\text{O}_2$ cells cycled under various voltage cutoff conditions at 0.5C.¹⁶⁰ Reproduced from ref. 160 with permission from Elsevier ref. 160 Copyright 2024.

Another critical challenge for layered oxide cathodes for Na- and K-ion batteries originates from the multi-phase transitions occurring during electrochemical charging and discharging, which are driven by strong alkali–alkali repulsion and changes in the alkali-ion coordination environments. In Na layered oxides, these transitions often manifest as steep and multi-step voltage profiles (Figure 8a).¹⁵⁹ These phase transitions are often more severe and irreversible, leading to structural collapse, loss of crystallinity, and long-term capacity fade for K-ion chemistries¹⁶¹⁻¹⁶³ The multiple phase transitions, resulting from the de/intercalation of large Na and K ions, lead to mechanical stress and microcracking, degrading the cycling performance.¹⁶⁴⁻¹⁶⁶ Ion doping and substitution strategies have been used to avoid such multiple phase transitions.¹⁶¹⁻¹⁷¹ For example, Ren *et al.*

developed $\text{P2-Na}_{0.67}\text{Li}_{0.08}\text{Mn}_{0.82}\text{Ti}_{0.1}\text{O}_2$, where Li and Ti are substituted into the distorted orthorhombic $\text{P'2-Na}_{0.67}\text{MnO}_2$.¹⁵⁹ The $\text{P2-Na}_{0.67}\text{Li}_{0.08}\text{Mn}_{0.82}\text{Ti}_{0.1}\text{O}_2$ exhibits phase-transition-free Na intercalation behavior, as shown in Figure 8a–b. The authors claimed that ion substitutions could suppress Na^+ /vacancy ordering with respect to the Na concentration, mitigating phase transitions upon charging–discharging. The phase-transition-free Na cathode exhibited significantly improved cycling stability (Figure 8c). However, the phase-transition-free Na intercalation is not always beneficial. Eliminating multiple voltage plateaus often produces an enhanced steep voltage curve, which could reduce the specific capacity within the given voltage window (Figure 8a). When a two-phase reaction exists at the end of charge or discharge, the voltage curve becomes flat, enabling more Na^+ -ion extraction or insertion for a given voltage limit. However, as discussed above, the multiple phase transition is detrimental to the capacity retention. Therefore, one plausible strategy is to improve the chemical stability of Na layered oxides at a high state of charge. In typical Na layered oxides, a higher cut-off voltage delivers higher specific capacity but poorer cycling performance (Figure 8d–f).¹⁶⁰ In high-voltage regions, several issues, including structure collapse, TM dissolution, and electrolyte decomposition, result in capacity degradation during cycling. Improving the chemical stability of Na layered oxides in high-voltage regions could be a viable yet challenging route to achieving high-capacity and high-energy-density Na-ion batteries.

curves of $\text{Na}_{4-x}\text{MnV}(\text{PO}_4)_3$ cathode material within 2.5–4.0 V voltage range.¹⁸⁸ Reproduced from ref. 188 with permission from American Chemical Society ref. 188 Copyright 2018. (d) Electrochemical charge and discharge profiles of $\text{Na}_3\text{V}_2(\text{PO}_4)_3$ (green) and c- $\text{Na}_2\text{V}_2(\text{PO}_4)_3$ (pink) electrodes.¹⁹² Reproduced from ref. 192 with permission from Springer Nature ref. 192 Copyright 2025.

Polyanion compounds offer a compelling alternative due to their robust 3D frameworks, which provide high structural and thermal stability and result in excellent cycling stability. As discussed in Section 3.1., polyanionic compounds exhibit much flatter voltage profiles than layered oxide compounds. The flatter voltage curves enable the de/intercalation of Na and K ions within a narrow voltage window, eliminating the requirement of a large voltage swing. In addition, the inductive effect of polyanion groups, such as phosphate or sulfate groups, enables elevated redox potentials for TM centers, improving the energy density.¹⁹³⁻¹⁹⁶ Given the aforementioned advantages, several polyanionic compounds have been developed, with a focus on V-, Fe-, and Mn-based compounds.^{150, 153, 172-190} Figure 9a plots the specific capacity vs. average discharge voltage of various polyanionic cathode compounds for Na- and K-ion batteries, categorized into compounds with V-, Fe-, and Mn-based redox centers.^{150, 153, 172-190} V-based polyanionic compounds, such as $\text{Na}_3\text{V}_2(\text{PO}_4)_3$, NaVOPO_4 , KVOPO_4 , and KVPO_4F , have been extensively studied due to their high redox potential and reasonably high specific capacity. However, existing electrolyte systems for Na- and K-ion batteries lack oxidative stability above 4.5 V. As a result, high-voltage V-based polyanionic materials experience rapid electrolyte decomposition and structural degradation, which in turn leads to poor cycling stability and limits their practical application.¹⁹⁷ Also, given the

earth abundance, price, and toxicity of vanadium,^{10, 11, 198} V-based polyanionic compounds may not be the best candidates for large-scale deployment. Fe- and Mn-based polyanionic chemistries may be more plausible options because of their abundance and price. However, Fe-based polyanionic compounds generally present much lower redox potential and specific energy than V- and Mn-based compounds, as highlighted in Figure 9a.

Mn-based compounds strike a good balance with their high redox potential and low cost. As a result, several Mn-based polyanionic compounds have been investigated, including $\text{Na}_4\text{MnV}(\text{PO}_4)_3$, $\text{Na}_4\text{MnCr}(\text{PO}_4)_3$, $\text{Na}_3\text{MnTi}(\text{PO}_4)_3$, and $\text{Na}_3\text{MnZr}(\text{PO}_4)_3$.^{188, 191, 199-201} Figure 9b shows the theoretical redox potential of $\text{Na}_4\text{MnCr}(\text{PO}_4)_3$, $\text{Na}_3\text{MnTi}(\text{PO}_4)_3$, and $\text{Na}_3\text{MnZr}(\text{PO}_4)_3$.¹⁹¹ In these compounds, the extraction of the last Na^+ ion is expected to be electrochemically inaccessible because the fully desodiated phases are neither chemically stable nor can current electrolyte systems survive at such a high voltage.^{191, 202-205} Therefore, $\text{Na}_3\text{MnTi}(\text{PO}_4)_3$ and $\text{Na}_3\text{MnZr}(\text{PO}_4)_3$ compounds can only utilize 2 Na^+ ion exchange unless accessing a low-voltage redox reaction of $\text{Ti}^{3+/4+}$. Utilizing the low-voltage $\text{Ti}^{3+/4+}$ redox is not viable for practical battery systems. Therefore, Wang *et al.* and Zhang *et al.* developed $\text{Na}_4\text{MnCr}(\text{PO}_4)_3$ as a potential Na-ion cathode candidate due to the potential to reversibly de/intercalate three Na^+ ions by utilizing the $\text{Mn}^{2+/3+/4+}$ and $\text{Cr}^{3+/4+}$ redox.^{190, 191} Importantly, $\text{Na}_4\text{MnCr}(\text{PO}_4)_3$ can deliver a high specific capacity up to 160 mAh g^{-1} with multi redox reactions of $\text{Mn}^{2+/3+}$, $\text{Mn}^{3+/4+}$, and $\text{Cr}^{3+/4+}$, as confirmed by X-ray absorption near-edge structure analysis.^{190, 191} Since then, several groups have investigated this material and optimized its performance.^{190, 206-215} However, several challenges remain: (i) a high upper cut-off voltage $\geq 4.6 \text{ V}$ is required, where liquid electrolytes are prone to decompose and (ii) an extremely low cut-off voltage for discharge is required, typically $< 1.5 \text{ V}$, to deliver a high capacity because of the slow kinetics at the end of

discharge. The low voltage cut-off could result in liquid electrolyte reduction at the cathode surface. In addition, such a low cut-off voltage limit is not useful for practical applications. When a reasonable lower cut-off voltage limit is used, the specific capacity could be reduced to 80-110 mAh g⁻¹.^{210, 212, 215}

Na₄MnV(PO₄)₃ has been considered as another potential Na-ion cathode material because of its excellent cycling performance and use of the low-cost Mn element.^{201, 216, 217} This cathode can exhibit a reasonably high capacity of ~100 mAh g⁻¹ by utilizing V^{3+/4+} and Mn^{2+/3+}. Interesting, Zakharkin *et al.* and Chen *et al.* found that the third Na⁺ ion could be extracted by utilizing the additional redox of V^{4+/5+} when the cut-off voltage increases over 4.0 V.^{188, 218} By doing so, the discharge capacity can be increased up to ~120 mAh g⁻¹. However, the extraction of the third Na⁺ ion from Na₄MnV(PO₄)₃ leads to an irreversible structure change, resulting in substantial discharge profile changes, in which plateaus no longer exist (Figure 9c).¹⁸⁸ Whereas the charging process from Na₃MnV(PO₄)₃ to Na₂MnV(PO₄)₃ follows a two-phase reaction, the discharge process from Na₂MnV(PO₄)₃ to Na₃MnV(PO₄)₃ follows a solid-solution reaction pathway once the third Na⁺ ion is extracted at a high voltage cut-off, which is related to the voltage profile changes.^{188, 218} In addition, it has been determined that the third Na extraction from Na₄MnV(PO₄)₃ results in accelerated capacity degradation upon cycling.¹⁸⁸ Stabilizing the third Na extraction process remains a critical challenge to utilize the full potential of the Na₄MnV(PO₄)₃ cathode.

As discussed above, the extraction of the last two Na⁺ ions from Na₄MnM'(PO₄)₃ (M' = V, Cr, Ti, Zr), followed by the reversible Na⁺ ion re-intercalation is extremely challenging. Although it is not the same composition, Park *et al.* recently proposed a new pathway to extract all the Na ions and intercalate back into the same structure type, NASICON.¹⁹² They identified the existence of a

metastable intermediate $\text{Na}_2\text{V}_2(\text{PO}_4)_3$ phase between $\text{Na}_3\text{V}_2(\text{PO}_4)_3$ and $\text{Na}_1\text{V}_2(\text{PO}_4)_3$ and successfully isolated this phase.^{192, 219, 220} Interestingly, two Na^+ ions could be deintercalated from and reinserted reversibly into the metastable $\text{Na}_2\text{V}_2(\text{PO}_4)_3$ phase with completely different voltage profiles compared to $\text{Na}_3\text{V}_2(\text{PO}_4)_3$ (Figure 9d).¹⁹² The metastable $\text{Na}_2\text{V}_2(\text{PO}_4)_3$ phase delivers a similar capacity as $\text{Na}_3\text{V}_2(\text{PO}_4)_3$; however, it is notable that the operating voltage increases substantially by utilizing $\text{V}^{3+/4+}$ and $\text{V}^{4+/5+}$, increasing the specific energy.

Fluorination has emerged as an effective strategy for enhancing the performance of polyanion-based cathode materials.²²¹⁻²²³ Substituting highly electronegative F^- ions into the polyanionic framework modifies the electron cloud distribution around the transition metal, thereby increasing the inductive effect and strengthening the ionic character of the metal–fluorine bonds.²²⁴ This structural and electronic modification raises the redox potential of the active transition metal, resulting in a higher operating voltage and consequently improving the overall energy density of the full cell.²²⁴ Furthermore, fluorination can also alter the sodium (de)insertion mechanism from a two-phase to a single-phase (solid-solution) reaction, which significantly reduces lattice strain and enhances structural stability during cycling.²²⁴ A few recent review articles^{224, 225} have provided in-depth discussions on fluorination in polyanionic cathodes, and readers are encouraged to consult these works for a more comprehensive understanding of its impacts on electrochemical performance.

3.3. Challenges and potential of Prussian blue analogue cathodes for Na- and K-ion batteries

PBAs have emerged as promising cathode materials for Na- and K-ion batteries owing to their open 3D framework, which allows for rapid Na- and K-ion diffusion and excellent cycling performance. Their low-cost, scalable synthesis from earth-abundant elements such as Fe and Mn makes them attractive for large-scale applications.

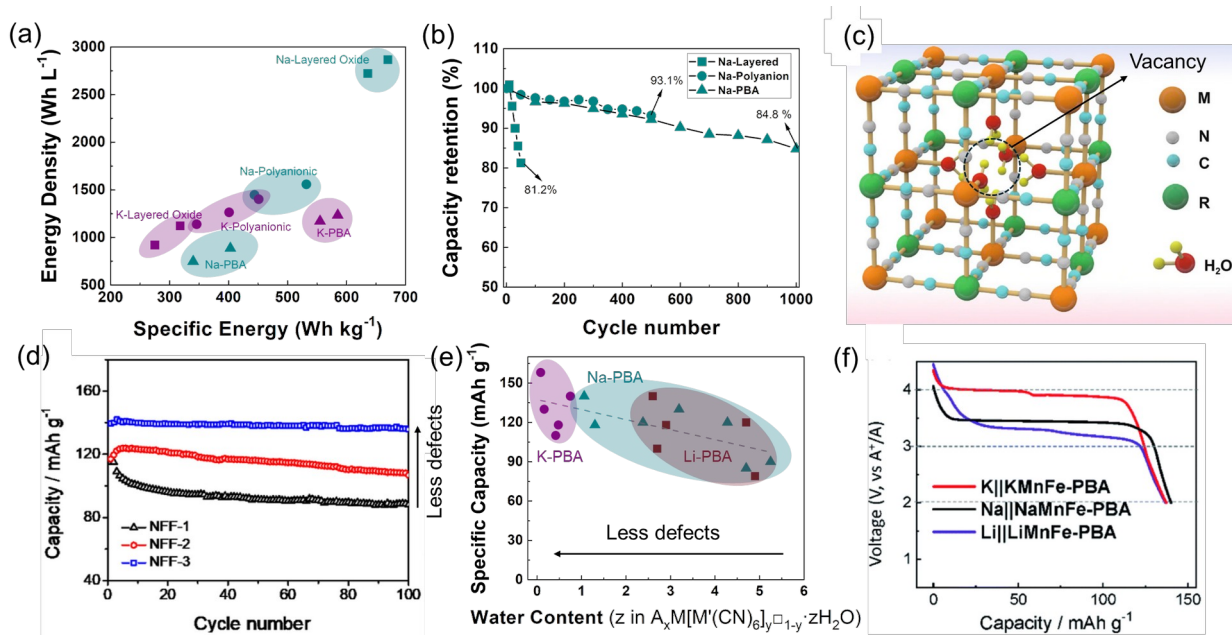


Figure 10. (a) Specific energy (Wh kg^{-1}) vs. energy density (Wh L^{-1}) of various cathode compounds for Na- and K-ion batteries.^{141, 142, 151-153, 156, 174, 185, 187, 191, 226-228} (b) Comparison of the capacity retention of various Na cathodes.^{141, 174, 228} (c) A defective $A_xM[R(CN)_6]$ framework with vacancies and interstitial water present in each unit cell.²²⁹ Reproduced from ref. 229 with permission from Springer Nature ref. 229 Copyright 2021. (d) Comparison of cycle performance of Na-enriched Prussian blue synthesized with 18 mmol NaCl (NFF-1), 12 mmol NaCl with 3 mmol trisodium citrate (NFF-2), and then 6 mmol trisodium citrate (NFF-3) at 0.2C in a range of 2.0–4.1 V.²³⁰ Reproduced from ref. 230 with permission from Elsevier ref. 230 Copyright 2020. (e) Water content vs. specific capacity of various Li-, Na-, and K-PBA compounds.^{106, 135, 231-236} In this

analysis, we used datapoints from the literature where the water molecule levels were quantified.

(f) Galvanostatic discharge curves of $\text{Li}_x\text{Mn}[\text{Fe}(\text{CN})_6]_y$ (LiMnFe–PBA), $\text{Na}_x\text{Mn}[\text{Fe}(\text{CN})_6]_y$ (NaMnFe–PBA), and $\text{K}_{1.75}\text{Mn}[\text{Fe}(\text{CN})_6]_{0.93}$ (KMnFe–PBA) in nonaqueous Li, Na, and K cells, respectively²³⁷ Reproduced from ref. 237 with permission from Royal Society of Chemistry ref. 237 Copyright 2022.

Considering the specific energy (Wh kg^{-1}) and energy density (Wh L^{-1}), PBAs would not be the best choice for Na-ion chemistries because these values are much lower for PBAs than for layered oxides and polyanionic compounds, as shown in Figure 10a.^{141, 142, 151-153, 156, 174, 185, 187, 191, 226-228} In the Na-ion system, the layered oxide compounds still deliver the highest level of specific energy and energy density compared to other types of candidates. However, to exhibit such high energy, the layered oxides must be cycled over wide voltage ranges, typically 4.5 to 1.5 V. Such a high voltage sweep often leads to undesired irreversible reactions, degrading the cycling performance (Figure 10b).²²⁸ In sharp contrast, polyanionic compounds and PBAs exhibit greatly improved cycling stability for Na-ion batteries.^{141, 174} For K-ion chemistries, it is obvious that PBAs and polyanionic compounds are a better choice compared with the layered oxides because these compounds exhibit higher specific energy and energy density (Figure 10a). Although polyanionic compounds often exhibit a slightly higher energy density than PBAs, a much higher specific energy density can be achieved by PBAs for K-ion batteries. Therefore, PBAs could be considered potential high-energy cathodes for K-ion batteries.

The low-to-mild temperature synthesis for PBAs is an important advantage for low-cost rechargeable batteries. However, such a low-temperature synthesis process often causes challenges

in controlling vacancy formation, interstitial water molecules, and crystal water molecules in the PBA framework (Figure 10c).²²⁹ In fact, these defects result in degradation of the electrochemical performance.²³⁸⁻²⁴¹ Therefore, several researchers have investigated strategies to reduce such defects and examined how these defects affect the electrochemical properties.^{230, 242-246} For example, Yan *et al.* synthesized a series of Na-enriched Fe-based PBAs ($\text{Na}_x\text{Fe}[\text{Fe}(\text{CN})_6]_{1-y} \cdot z\text{H}_2\text{O}$) with varied concentrations of $[\text{Fe}(\text{CN})_6]$ vacancies and coordinated water and investigated the effects of the lattice defects on the reversible capacities and cycling stabilities.²³⁰ As shown in Figure 10d, PBAs with lower lattice defects deliver higher specific capacity and improved cycling performance. Similarly, a study by Want *et al.* confirmed the detrimental effects of lattice defects in PBAs.²⁴⁶ Our analysis also confirmed the similar trend that higher water content in PBA compounds, in general, reduces the specific capacity in Li-, Na-, and K-ion chemistries (Figure 10e).^{106, 135, 231-235} A recent study by Ge and colleagues demonstrated that the removal of interstitial water molecules from PBAs is critical to improving their electrochemical performance; however, eliminating both interstitial water and coordination water molecules completely could collapse the PBA framework and degrade the electrochemical performance.²⁴⁴ This finding indicates that the careful control of lattice defects, including interstitial water and coordination water molecules, is required for optimization of PBA cathodes. In recent years, a few studies have claimed that K-based PBAs usually contain a lower H_2O content than Na-based PBAs.²⁴⁷⁻²⁵⁰ Similarly, our analysis using datapoints from published studies confirmed that K-PBAs generally possess lower water content in the PBA framework compared with Na and Li counterparts (Figure 10e).^{95, 124, 217-221} Given the critical role of interstitial water molecules in the PBA framework, K-PBA could show more promise compared with Na analogues, and K doping could be a beneficial strategy in improving the electrochemical performance of Na-PBAs. Indeed, Gao *et al.* investigated the K-doping strategy in

Na-PBAs and found that the introduction of K ions decreases the interstitial water molecule content while improving the stability of PBAs.²⁴⁹ These authors claimed that the large K^+ ion could reduce the occupancy possibility of interstitial water molecules in the PBA framework. More recently, Xu and colleagues studied the effect of K^+ -ion substitution into Na-PBAs.²⁴⁵ These researchers found that the water content in PBAs linearly decreases as the K/Na ratio increases. Their density functional theory calculations showed that dehydration from Na-PBA requires four times higher energy than that for the dehydration from K-PBA. This finding indicates that the dehydration of K-PBA compounds is much easier than for their Na counterparts. In addition, these authors found that the K substitution not only enhances the thermal stability of PBAs but also restrains HCN generation, which might be attributable to their lower water content. Lower HCN generation could contribute to improved safety.¹⁰⁷ In fact, toxic cyanide gases evolving from PBAs could be a potential safety hazard.^{107, 251} A generated toxic cyanide gas species can react exothermically with the organic electrolyte, propagating thermal runaway through an oxygen-free route.^{252, 253}

Another important advantage of PBAs in K-ion chemistries is highlighted when the operating voltage is compared among Li-, Na-, and K-ion intercalation. The K-intercalation voltage in PBA compounds is typically higher than for their Li and Na counterparts, resulting in higher specific energy (Figure 10f).²³⁷ This finding is likely attributable to the fact that the insertion of a large K^+ ion into the large cavity in the PBA framework stabilizes the discharged state compared to the insertion of smaller ions such as Na^+ and Li^+ . Intriguingly, the smaller Li^+ intercalation voltage is often lower than the Na^+ intercalation voltage in PBA compounds (Figure 10f).²³⁷

One of the major concerns around PBAs is their intrinsically low tap density, which is a factor that can substantially limit the volumetric energy density achievable in a full battery configuration.²⁵⁴

For improving the tap density of PBAs, strategies such as morphology control through recrystallization²⁵⁵ and spherical particle design²⁵⁴, chelating agent-assisted synthesis¹⁰⁶, potassium-ion-assisted strategies²⁵¹, mechanochemical synthesis²⁵⁶, and multi-component doping²⁵⁷ etc. have been introduced. Zheng *et al.* demonstrated that utilizing a dual-vortex coprecipitation chamber enables the controlled synthesis of spherical agglomerate PBAs, which significantly improves tap density to 1.60 g cm⁻³ and optimizes particle packing within the electrode.²⁵⁴ Building on these advances, Fan *et al.* introduced a recrystallization-driven strategy to produce monoclinic binary hexacyanoferrate with quasi-spherical particles and high crystallinity, achieving a tap density of 0.992 g cm⁻³ and facilitating effective electrode compaction for commercial applications.²⁵⁵

4. Summary and outlook

As the demand for energy storage increases, the development of lower-cost, earth-abundant rechargeable batteries has become a central topic globally. As discussed in Section 1, traditional LIBs, which rely on conventional Co- and Ni-based layered oxide cathodes, cannot meet this surging demand (Figure 1). Therefore, a paradigm shift is required to develop cathode materials that can eliminate the use of Co and Ni. DRX compounds have attracted significant attention because they can be synthesized without the use of these two critical elements.^{22, 29} Although understanding and optimization of the compositional tuning of DRX compounds remain important tasks, partially due to their high compositional tunability that accommodates versatile TMs, a wide range of Li-excess levels, and fluorine substitutions, we expect that the most critical step is to advance the processability of the DRX positive electrode fabrication and their evaluation in close-to-practical environments. Currently, most DRX studies continue to use high-energy ball milling or shaker

milling to reduce the particle size and create homogeneous mixing with carbon additives.^{45, 57, 59, 61, 81-87} In addition, DRX compounds have been evaluated with 10–30 wt% carbon additive and relatively low loading density.^{45,48, 57, 59, 61, 81-87} These evaluations are, in fact, far from practical. It is therefore necessary to evaluate DRX positive electrodes in more practical environments and develop new methodologies to fabricate DRX electrodes with a significantly lower inactive component (carbon and binder) content and higher loading without compromising performance.

Na- and K-ion batteries are viable solutions for the development of low-cost, earth-abundant rechargeable batteries. However, given the growth of the LiFePO₄/graphite battery system and its commercialization,²⁵⁸ newly developed Na- and K-ion batteries must be competitive with LiFePO₄-based LIBs in terms of cost and energy density. Therefore, Mn- and Fe-based cathode development is urgent, although V-based cathode materials such as Na₃V₂(PO₄)₃ and KVPO₄F exhibit excellent electrochemical performance.¹⁸³⁻¹⁸⁷ For Na-ion chemistries, layered oxide compounds have significant advantages in terms of specific energy and energy density over other candidates, including polyanionic compounds and PBAs (Figure 9a). The remaining important quest is to improve their cycling performance without compromising their energy density. Polyanionic compounds may be another viable route; however, their energy density must be improved. For K-ion batteries, we anticipate that polyanionic compounds and PBAs will be more compelling candidates than layered oxides, in contrast to the Li- and Na-ion systems. However, studies on K polyanionic compounds need to be shifted to explore Fe- and Mn-based compounds with higher energy density.

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Author contributions

H. K. and H.L. conducted the literature survey and analysis and wrote the original draft. V.S.A. and S.J. contributed to the data analysis from reported articles and visualization. All the authors reviewed and edited the final draft.

Data availability

No primary research results have been included, and no new data were generated as part of this review article.

Conflict of Interest

The authors declare no conflict of interest.

Declaration of AI Assistance in Writing

The authors utilized ChatGPT to assist with paraphrasing, grammar correction, language polishing, and enhancing overall coherence. They carefully reviewed and edited all content to guarantee its accuracy and originality, and they take full responsibility for the final version of the publication.

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