



Review

Recent developments in cathode materials for lithium ion batteries

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ABSTRACT

One of the challenges for improving the performance of lithium ion batteries to meet increasingly demanding requirements for energy storage is the development of suitable cathode materials. Cathode materials must be able to accept and release lithium ions repeatedly (for recharging) and quickly (for high current). Transition metal oxides based on the α - NaFeO_2 , spinel and olivine structures have shown promise, but improvements are needed to reduce cost and extend effective lifetime. In this paper, recent developments in cathode materials for lithium ion batteries are reviewed. This includes comparison of the performance characteristics of the promising cathode materials and approaches for improving their performances.

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1. Introduction

The development of improved battery technology is critical for advancements in a variety of applications ranging from hybrid electric vehicles to consumer electronics [1,2], and improved battery performance depends on the development of materials for the various battery components [3–6]. Most lithium ion batteries use organic solvents as the electrolyte, the most common being LiPF_6 , which has a low electrical resistance [7], and is typically mixed with carbonates. Solid electrolytes, including polymers [8] and inorganic compounds [9,10], are used for solid state batteries, which have advantages in terms of miniaturization and durability. The most common anode materials are carbon-based compounds and lithium-containing alloys. Both approaches result in the establish-

ment of a reduced lithium activity (as compared to lithium metal), which reduces reactivity with the electrolyte and improves safety, but also leads to a lower cell voltage. There are efforts in the development of improved electrolyte and anode materials, but the focus of this paper is on the cathode materials.

Cathode materials are typically oxides of transition metals, which can undergo oxidation to higher valences when lithium is removed [11,12]. While oxidation of the transition metal can maintain charge neutrality in the compound, large compositional changes often lead to phase changes, so crystal structures that are stable over wide ranges of composition must be used. This structural stability is a particular challenge during charging when most (ideally all) of the lithium is removed from the cathode. During discharge lithium is inserted into the cathode material and electrons from the anode reduce the transition metal ions in the cathode to a lower valence. The rates of these two processes, as well as access of the lithium ions in the electrolyte to the electrode surface, control the maximum discharge current. Exchange of lithium ions with the

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electrolyte occurs at the electrode–electrolyte interface, so cathode performance depends critically on the electrode microstructure and morphology, as well as the inherent electrochemical properties of the cathode material. For example, there is considerable work on the use of nanostructured electrodes with high surface and interfacial areas to improve performance [13–17]. While this paper will include some discussion of general microstructural features, the focus is on the cathode materials rather than the microstructures.

2. Cathode materials

The cathode material most commonly used in lithium ion batteries is LiCoO_2 [18]. LiCoO_2 forms the $\alpha\text{-NaFeO}_2$ structure, which is a distorted rock-salt structure where the cations order in alternating (1 1 1) planes. This ordering results in a trigonal structure ($R\bar{3}m$) and, for LiCoO_2 , planes of lithium ions through which lithiation and delithiation can occur [19]. Although LiCoO_2 is a successful cathode material, alternatives are being developed to lower cost and improve stability. Cobalt is less available, and thus more costly, than other transition metals, such as manganese, nickel and iron. In addition, LiCoO_2 is not as stable as other potential electrode materials and can undergo performance degradation or failure when overcharged [20–22]. The increase in charging voltage can increase the cell capacity, but can also lead to more rapid decrease in capacity as the cell is cycled during recharging [23]. Several reasons have been given for the degradation during cycling. One is that cobalt is dissolved in the electrolyte when the electrode is delithiated during charging [24], such that less lithium can be intercalated during discharge. Another is that the CoO_2 layer formed after full delithiation shears from the electrode surface [25], which also results in less capacity for lithium intercalation. In addition, there is a sharp change in lattice parameter with change in lithium content [26], which can lead to stresses and micro-cracking of the cathode particles [27]. Stoichiometric LiCoO_2 can be difficult to obtain [28], so heat treatment to control the surface phase content is needed to improve performance during cycling [29].

LiNiO_2 , which also forms the $\alpha\text{-NaFeO}_2$ structure, is lower in cost and has a higher energy density (15% higher by volume, 20% higher by weight) [30], but is less stable [31,32] and less ordered [33], as compared to LiCoO_2 . The lower degree of ordering results in nickel ions occupying sites in the lithium plane, which impedes lithiation/delithiation and also creates challenges in obtaining the appropriate composition [34]. The addition of cobalt to LiNiO_2 increases the degree of ordering, which leads to nickel ions occupying sites in the nickel/cobalt plane rather than in the lithium plane [12]. Thus, $\text{LiNi}_{1-x}\text{Co}_x\text{O}_2$, typically containing mostly nickel ($x \sim 0.8$), has been used to take advantage of the low cost and higher capacity of nickel relative to cobalt [35–39]. LiMnO_2 forms a monoclinic, rather than rhombohedral, structure [40], which can transform to a layered hexagonal structure during cycling [41]. The addition of nickel [42,43], or more commonly nickel and cobalt, to LiMnO_2 can lead to the formation of the $\alpha\text{-NaFeO}_2$ structure [44,45]. The ratio of the trigonal lattice parameters, c/a , depends on the composition, and as this ratio approaches 1.633 the distortion from cubic symmetry decreases, which leads to less ordering and thus more transition metal ions in the lithium ion plane [12]. The most commonly used $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ composition contains equal amounts of the three transition metals, i.e. $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$, and has high capacity [46], good rate capability [47,48] and can operate at high voltages. A higher charging voltage increases the capacity, but also leads to more rapid loss of capacity during cycling [49–51]. As with $\text{LiNi}_{1-x}\text{Co}_x\text{O}_2$, cobalt helps to reduce the amount of nickel in the lithium layer [12] and small amounts of cobalt (up to 0.20–0.25) have been shown to improve capacity [51–57]. Increased cobalt content can also reduce the loss in capac-

ity during cycling [51,58–60]. The improved performance has been attributed to cobalt increasing the conductivity [51,59] and improving the structural stability [59,60] of the cathode. Although nickel in the lithium layer can be detrimental to lithium transport, it has been shown to stabilize the structure during delithiation and thus improve cycling performance [61]. $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ can be overlithiated, which has been shown to improve electrode performance [62,63], especially with low cobalt contents [64]. There can be an oxidative loss during the first cycle, which is not recovered during normal cycling [65]. However, deep discharging has been shown to recover this first-cycle loss [66].

Another promising cathode material is LiMn_2O_4 that forms a spinel structure ($Fd\bar{3}m$), in which manganese occupies the octahedral sites and lithium predominantly occupies the tetrahedral sites [67]. In this case, the paths for lithiation and delithiation are a 3-dimensional network of channels rather than planes, as in the $\alpha\text{-NaFeO}_2$ structure. LiMn_2O_4 is lower cost and safer than LiCoO_2 [12,68,69], but has a lower capacity as compared to the cathode materials that form the $\alpha\text{-NaFeO}_2$ structure described above [46,70]. One of the challenges in the use of LiMn_2O_4 as a cathode material is that phase changes can occur during cycling [71–73]. For example, LiMn_2O_4 cathodes have been field tested in the DC power supply of an operating telecommunications transceiver. During this test, a relatively rapid loss of capacity occurred in the first few days, but the rate of capacity loss subsequently decreased [74]. The initial loss has been attributed to loss of oxygen during charging [75]. Capacity loss has also been observed during storage due to dissolution of manganese in the electrolyte [76], or due to changes in particle morphology or crystallinity [77,78]. Other transition metals, including iron [79] and cobalt [80–85], and have been added to LiMn_2O_4 . The addition of iron results in an additional discharge plateau at high voltages, while cobalt improves the capacity retention during cycling by stabilizing the spinel crystal structure. However, the most common addition to LiMn_2O_4 is nickel [86], which decreases the lattice parameter and the electrical conductivity of LiMn_2O_4 [87]. The capacity increases with increasing manganese content and a 3:1 Mn:Ni ratio (i.e. $\text{Mn}_{1.5}\text{Ni}_{0.5}\text{O}_4$) is the most commonly used composition [88,89]. The manganese and nickel cations can order on the octahedral sublattice, but a disordered spinel structure has been shown to have a higher capacity [90]. Partial substitution of cobalt for nickel (i.e. $\text{Li}[\text{Mn}_{1.42}\text{Ni}_{0.42}\text{Co}_{0.16}]\text{O}_4$) has been used to reduce the formation of $\text{Li}_x\text{Ni}_{1-x}\text{O}$, which can degrade cell performance during cycling [91]. The addition of nickel to the surface of LiMn_2O_4 through coatings, rather than as a bulk dopant, can also be effective in improving capacity retention during cycling [92–94].

Vanadium oxide forms layered compounds and vanadium can have multiple valences, so vanadium oxides have been used as electrode materials. In particular, orthorhombic V_2O_5 [95–99] and monoclinic LiV_3O_8 [100–104] have been used as cathode materials. These electrodes have high capacities, but relatively low voltages (typically 3 V or less) as compared to the compounds discussed above.

Another promising class of cathode materials are phosphates (LiMPO_4) with the olivine structure ($Pnma$), in which phosphorous occupies tetrahedral sites, the transition metal (M) occupies octahedral sites and lithium forms one-dimensional chains along the [0 1 0] direction [105]. The phosphate most commonly used for the cathode is LiFePO_4 , which delithiates to FePO_4 as the Fe^{2+} is oxidized to Fe^{3+} [106]. Some iron ions occupy lithium sites, which results in the formation of lithium ion vacancies to maintain charge neutrality [107–109]. There is a miscibility gap between FePO_4 and LiFePO_4 [110], so the delithiation occurs by growth of a two-phase front rather than a continuous change in lithium content [111–116]. The formation of a two-phase mixture establishes a fixed activity, which results in a relative flat discharge profile (i.e. the voltage

remains relatively constant during discharge) [46]. Electronic conduction in LiFePO_4 occurs by small polaron hopping [117] and is relatively low ($10^{-9} \text{ S cm}^{-1}$ for pure LiFePO_4 [12]). Conductivity can be improved by heat treating to increase the hole conductivity [118], but the addition of a conductive phase is generally needed for satisfactory performance [13]. Additives for increasing the conductivity of LiFePO_4 will be discussed below, but one conductive phase, Fe_2P , can form during preparation and/or use and has been observed to improve performance [119,120], so Fe_2P is sometimes deliberately added in $\text{LiFePO}_4/\text{Fe}_2\text{P}$ composites [121–124]. The amount of Fe_2P is critical because small amounts increase conductivity, but larger amounts block lithium ion paths [125].

Other phosphates used for cathodes in lithium ion batteries include LiMnPO_4 [126–128] and LiCoPO_4 [128,129]. LiMnPO_4 and LiCoPO_4 have higher open circuit voltages (4.1 and 4.8 V, respectively) than LiFePO_4 (3.5 V) [12], but have lower capacities. For example, the capacities of LiMnPO_4 and LiCoPO_4 prepared by microwave hydrothermal synthesis were reported to be $\sim 1/6$ and $\sim 1/3$, respectively, that of LiFePO_4 prepared by the same process [128]. In addition, $\text{Mn}_2\text{P}_4\text{O}_7$ and $\text{Co}_2\text{P}_4\text{O}_7$ have been observed to form in delithiated LiMnPO_4 [127] and LiCoPO_4 [129] electrodes, respectively, which degrades the lifetime and can be a safety concern as oxygen is evolved during the decomposition reaction. Mixtures of phosphates, including LiMnPO_4 [130–132] or LiCoPO_4 [133,134] with LiFePO_4 , have been used for cathode materials. In such mixtures, the operating voltage increases with increasing manganese content [135,136], while capacity increases with increasing iron content [133,134,137]. Although LiNiPO_4 also forms the olivine structure [105] it is not typically used as a cathode material. However, nickel has been added to other phosphate cathode materials, including simple lithium transition metal phosphates (e.g. LiFePO_4 [138,139] and LiMnPO_4 [140]), and complex compounds, (e.g. $\text{Li}(\text{Mn,Fe})\text{PO}_4$ [141,142] and $\text{Li}(\text{Mn,Fe,Co})\text{PO}_4$ [143,144]). Another phosphate used as a cathode is $\text{Li}_3\text{V}_2(\text{PO}_4)_3$, which forms a monoclinic structure ($P2_1/n$) [145,146]. $\text{Li}_3\text{V}_2(\text{PO}_4)_3$ has a high operating voltage and good performance at high discharge currents [147,148]. For example, plateau voltages of greater than 4 V for discharge currents of 0.2–2C and greater than 3.9 V for a discharge current of 10C have been reported [148]. Vanadium additions have also been shown to improve the capacity of LiFePO_4 , especially at high discharge currents [138,149,150]. For example, at 0.1C, the increases in capacity are relatively modest (5–15%) [138,149,150], while at 10C increases in capacity of 80% to more than 200% have been observed [149,150]. The beneficial effect of vanadium additions has been attributed to enhancing lithium diffusion [138,150] or reducing the energy required for nucleation of LiFePO_4 in the $\text{LiFePO}_4\text{--FePO}_4$ [149] two-phase region.

3. Cathode performance

The multitude of materials, geometries and operational variables in lithium ion batteries complicates comparison of the performances of different cathode materials. Although there are a few reports in which different types of electrodes are tested in the same conditions and compared on a single plot (e.g. [47]) most reports focus on a particular type of electrode material with variations in composition or microstructure. On the other hand, summaries of results from different sources (e.g. [151]) may include results for different operating conditions, which complicates making comparisons between materials. The voltage ranges for different electrodes have been summarized [152], but this summary does not include capacities.

In this paper, the performances of different cathodes will be summarized according to operating conditions, so that comparisons can be made between results from different sources. Results

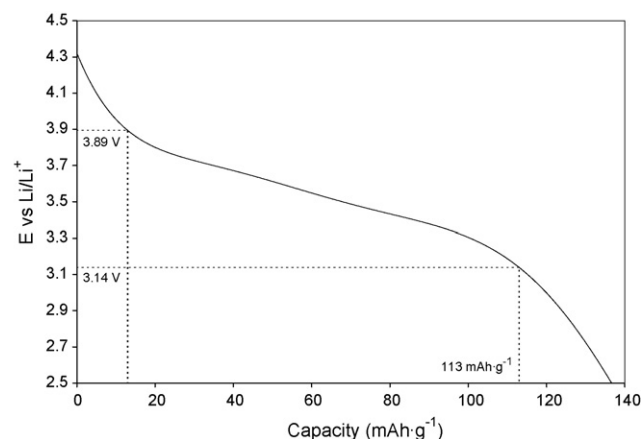


Fig. 1. Schematic discharge curve for lithium ion battery with parameters used in Figs. 2–5.

for different electrodes will be included on the same plots or on different plots with the same scales to allow for direct comparison between materials. In addition, multiple results for each electrode will be included to reflect the variations between reports, which can occur due to, for example, microstructural or morphological differences. To make such comparisons, the voltage range and capacity for each electrode have been determined as shown in Fig. 1. The voltage range is determined by the points of inflection in the discharge curve and the capacity is the capacity at second point of inflection. The capacities determined in this way (Figs. 2–5) are lower than the corresponding maximum capacities, but this method eliminates the lower cut-off voltage as a variable and allows for comparison of results from different sources. The purpose is not to present the absolute maximum capacity, but rather to provide a comparison of the useful voltage/capacity ranges of the voltage plateau for operation. The subsequent plots (Figs. 6–12), however, showing the trends in capacity with discharge current and after cycling use the maximum capacities reported in the respective articles.

The operating voltages and capacities of LiFePO_4 [123,153–162] and LiCoO_2 [163–167] with a charging voltage of 4.2 V and discharge current of 1C are shown in Fig. 2. The operating voltage for LiCoO_2 is higher than that for LiFePO_4 and LiFePO_4 has a narrower voltage range. The narrow voltage range for LiFePO_4 is a result of the formation of a two-phase mixture, rather than a continuous change in lithium content. Fig. 3 shows that a reduction in the discharge current by a factor of 10 (i.e. 0.1C) increases

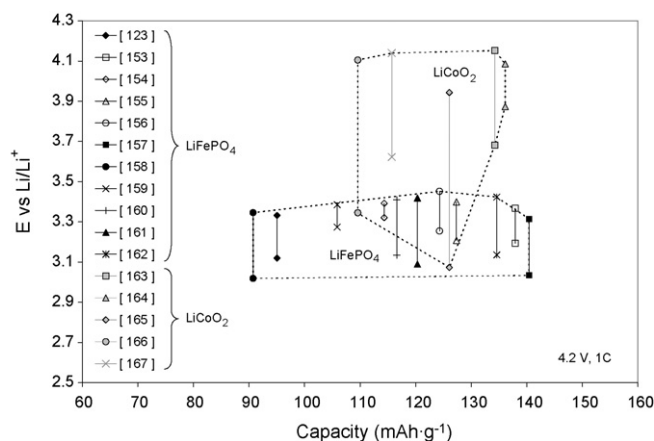


Fig. 2. Plateau voltage and capacity (see Fig. 1) for LiFePO_4 [123,153–162] and LiCoO_2 [163–167] with a charging voltage of 4.2 V and discharge current of 1C.

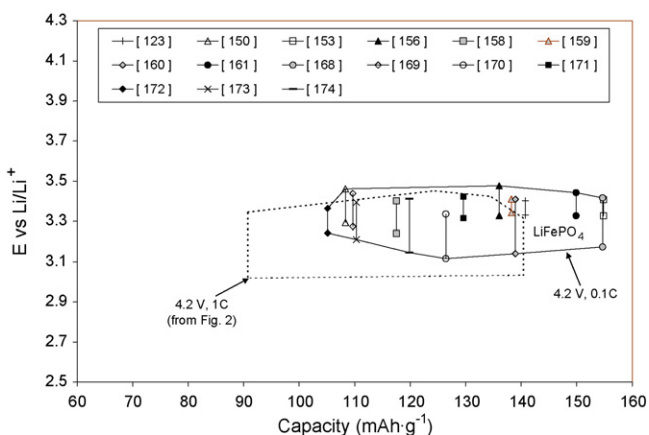


Fig. 3. Plateau voltage and capacity (see Fig. 1) for LiFePO_4 [123,150,153,156,158–161,168–174] with a charging voltage of 4.2 V and discharge current of 0.1 C.

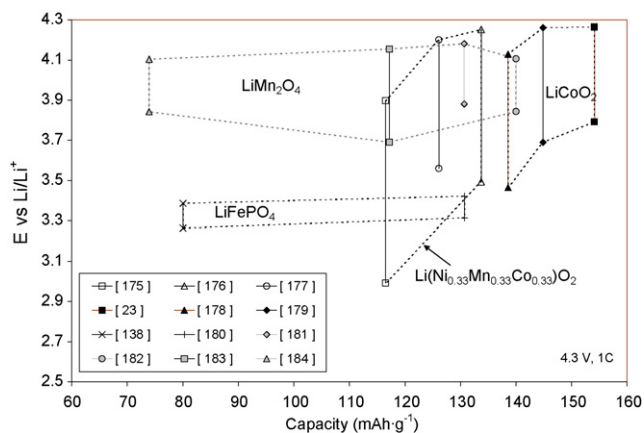


Fig. 4. Plateau voltage and capacity (see Fig. 1) for $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [175–177], LiCoO_2 [23,178,179], LiFePO_4 [138,180] and LiMn_2O_4 [181–184] with a charging voltage of 4.3 V and discharge current of 1 C.

the capacity of LiFePO_4 [123,150,153,156,158–161,168–174]. Figs. 4 and 5 summarize the operating voltages and capacities for $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [63,175–177], LiCoO_2 [23,178,179,189], LiFePO_4 [138,180] and LiMn_2O_4 [181–188] at a higher charging voltage of 4.3 V. LiMn_2O_4 , LiCoO_2 and $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ all

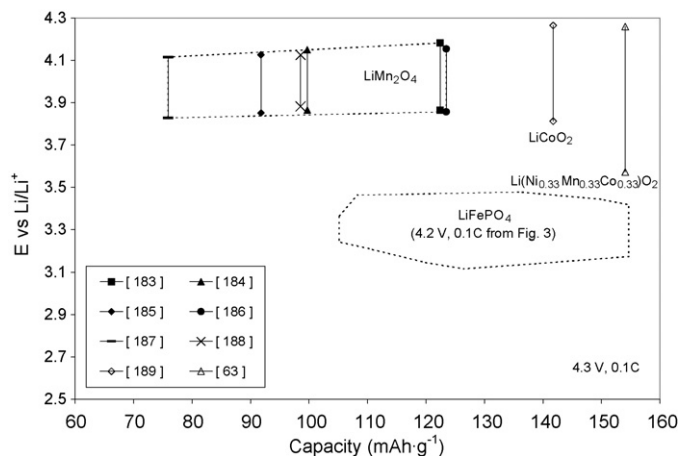


Fig. 5. Plateau voltage and capacity (see Fig. 1) for $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [63], LiMn_2O_4 [183–188] and LiCoO_2 [189] with a charging voltage of 4.3 V and discharge current of 0.1 C.

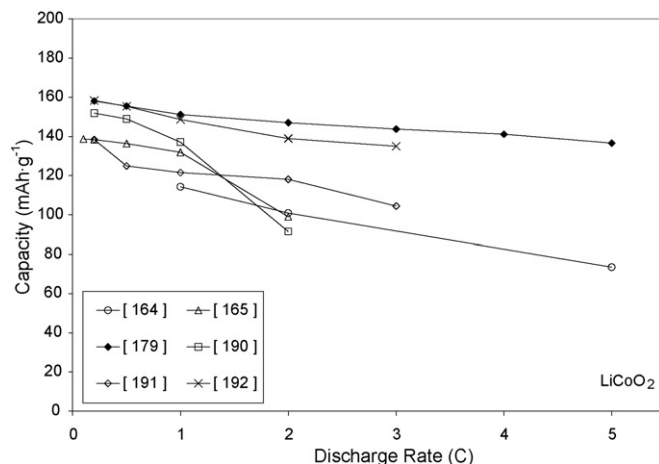


Fig. 6. Discharge capacity of LiCoO_2 as a function of discharge rate [164,165,179,190–192].

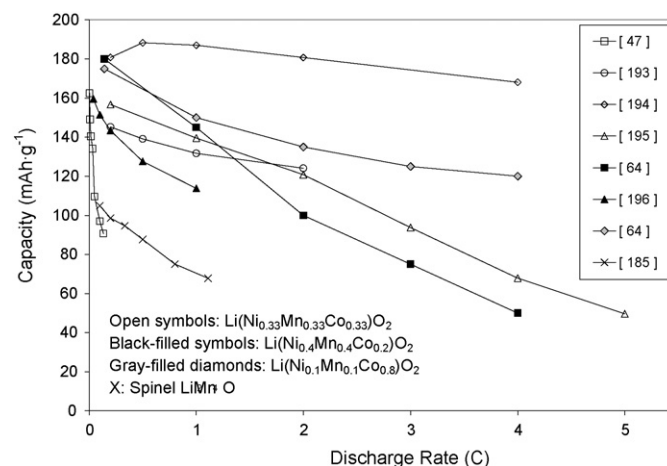


Fig. 7. Discharge capacity of $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ [47,64,193–196] and LiMn_2O_4 [185] as a function of discharge rate.

have higher operating voltages as compared to LiFePO_4 . LiMn_2O_4 has a similar, or higher, operating voltage as compared to LiCoO_2 and $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$, but its capacity is lower. Although the number of data points is small, with a decrease in discharge current from 1 to 0.1 C (compare Figs. 4 and 5), the capacity of

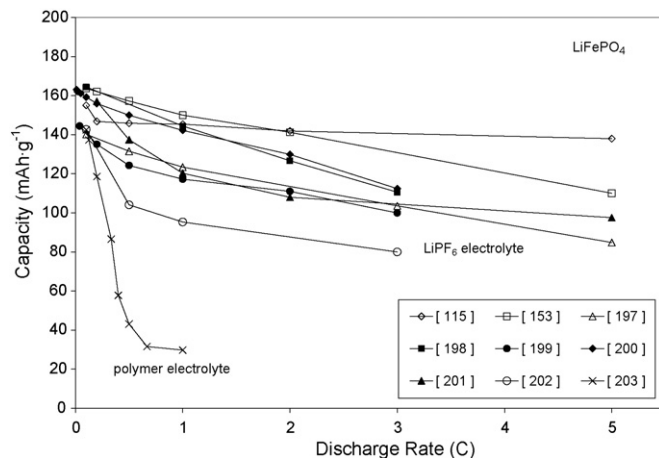


Fig. 8. Discharge capacity of LiFePO_4 as a function of discharge rate [115,153,197–203].

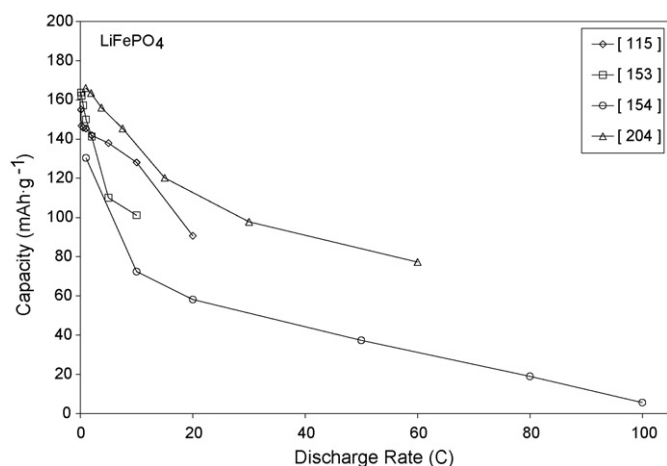


Fig. 9. Discharge capacity of LiFePO₄ at high discharge currents [115,153,154,204].

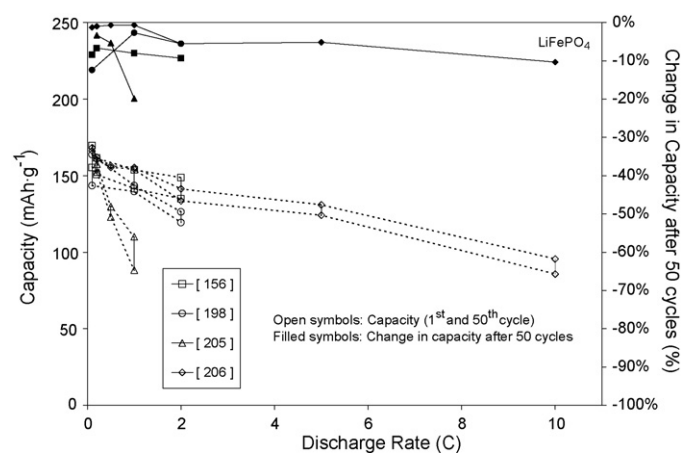


Fig. 10. Capacity after 1st/50th cycle and percent change in capacity after 50 cycles for LiFePO₄ cathodes [156,198,205,206].

Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ increases more than that of LiCoO₂, suggesting that the kinetics of charge transfer and/or mass transport are slower in Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ than in LiCoO₂.

The trend with discharge current is also illustrated in Figs. 6 and 7, which show the capacity as a function of discharge current for LiCoO₂ [164,165,179,190–192], Li(Ni,Mn,Co)O₂ [47,64,193–196] and LiMn₂O₄ [185]. Although there is significant variation among the results, the decrease in capacity with increas-

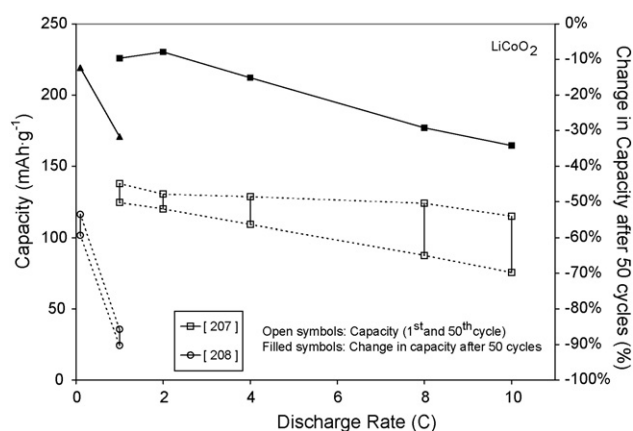


Fig. 11. Capacity after 1st/50th cycle and percent change in capacity after 50 cycles for LiCoO₂ cathodes [207,208].

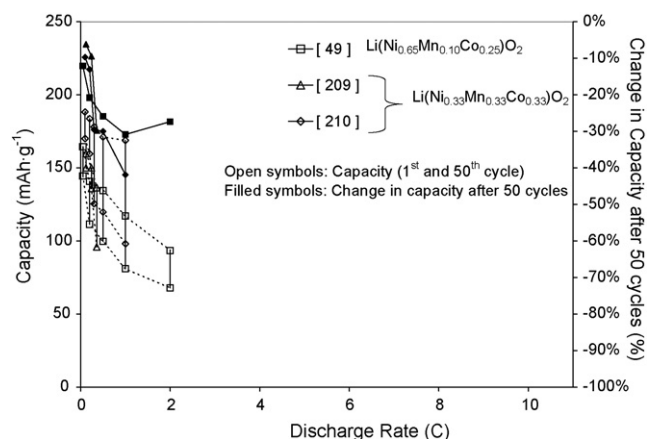


Fig. 12. Capacity after 1st/50th cycle and percent change in capacity after 50 cycles for Li(Ni,Mn,Co)O₂ cathodes [49,209,210].

ing discharge current is generally smaller for LiCoO₂ than for Li(Ni,Mn,Co)O₂. Fig. 8 shows that the discharge rate dependence of capacity for LiFePO₄ is similar to that for LiCoO₂. The result exhibiting a rapid decrease in capacity (symbol “x” in Fig. 8) is for a cell with a polymer electrolyte, rather than a liquid LiPF₆-based electrolyte, so the high current performance may be limited by the electrolyte rather than the electrode. Fig. 9 shows that LiFePO₄ can be used at high discharge currents.

The change in performance during cycling for LiFePO₄ [156,198,205,206], LiCoO₂ [207,208], Li(Ni,Mn,Co)O₂ [49,209,210] are compared in Figs. 10–12 by plotting the capacities after the 1st and 50th cycles as a function of discharge current. In addition, the right axis is used to show the percent change in capacity during the 50 cycles. The decrease in capacity of LiFePO₄ (~10–20%) after cycling is much smaller than that for LiCoO₂ or Li(Ni,Mn,Co)O₂ (~30–40%). These data also illustrate the sharper decrease in capacity with increasing discharge rate for Li(Ni,Mn,Co)O₂ as discussed above.

4. Composite cathodes

The combination of two electrode materials to form a composite electrode can be used to improve performance [211]. For example, the addition of LiFePO₄ to other electrodes, including LiCoO₂ [178,212], Li(Li_{0.17}Mn_{0.58}Ni_{0.25})O₂ [212] and Li(Ni_{0.5}Mn_{0.3}Co_{0.2})O₂ [213], improves capacity retention during cycling and performance at high discharge currents. Similarly, a phosphate surface treatment can improve capacity [214] or performance after cycling [215] of oxide electrodes. Monoclinic (C2/m) Li₂MnO₃ acts as a lithium reserve and improves capacity retention during cycling of layered LiMO₂ cathode materials, including Li(Co_{1-y}Ni_y)O₂ [216], Ni_{0.8}Co_{0.15}Zr_{0.05}O₂ [217], LiNi_{0.5}Mn_{0.5}O₂ [218], Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ [218–221] and other Li(Ni,Mn,Co)O₂ compositions [218,222–224]. Spinel electrodes have been combined with layered cathode materials, including LiCoO₂ [225] and Li(Ni,Mn,Co)O₂ [226,227] to expand the operating voltage range. The lithium is removed from the spinel at high voltages and then from Li(Ni,Mn,Co)O₂ at lower voltages [226].

The interface between the two components is where charge and mass transfer occur and is thus important in the operation of the electrode, so mechanical activation [225] or heat treatment [227] has been used to take full advantage of composite properties. LiCoO₂ and Li(Ni,Mn,Co)O₂ have been combined in a composite electrode [228], in which case the improved performance was attributed to the microstructure (i.e. combination of large and small particles) rather than to the inherent electrochemical properties of

the cathode materials. This illustrates one of the challenges in comparing electrode materials, in that the performance of the cathode, particular in a two-phase material, depends on the morphology and geometry of its constituents.

Composite cathodes can also be formed by coating particles of one cathode material with another active material rather than mixing separate particles. For example, the cycling and rate capability of LiMn_2O_4 has been improved with a Co_3O_4 coating, which is purported to form a fluoride layer and reduce electrode degradation [229]. Another example is the reduction in the capacity loss during cycling of LiNiO_2 with a cobalt–manganese coating, which has been attributed to suppression of a detrimental phase transition [230]. Vanadium compounds, including V_2O_5 [231] and LiV_3O_8 [223], which have high capacities, but relatively low operating voltages, have been used to increase the capacity of $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ by providing supplemental capacity at lower voltages late in the discharge cycle. Another lower voltage electrode that has been used in composite cathodes is $\text{Li}_4\text{Ti}_5\text{O}_{12}$. Because of its low voltage (~ 1.5 V vs. Li/Li^+), $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is more commonly used as an anode material, but it can be used as the cathode in low voltage cells [232–236]. $\text{Li}_4\text{Ti}_5\text{O}_{12}$ coatings have been shown to improve the capacity of LiCoO_2 [237] and LiMn_2O_4 [238] cathodes, as well as to improve the capacity retention during cycling of $\text{LiMn}_{1.4}\text{Cr}_{0.2}\text{Ni}_{0.4}\text{O}_4$ [239]. Similarly, coating LiCoO_2 with a lithium-conducting solid electrolyte material (LiPON) has also been used to improve the capacity and cycling performance of the cell [240] presumably by enhancing the kinetics and mass transfer at the electrode interface.

5. Effect of doping

The performance of cathode materials can be improved by doping, but the interpretation of doping effects can be complicated by the interrelations between doping and microstructure and morphology, since the microstructure formed can be affected by the dopant additions. Some examples in which the effects of doping on the electrochemical properties of the electrode are attributed to the effects of the dopant on the cathode microstructure or morphology rather than the effects on the material properties include cesium-doping of LiMn_2O_4 [241], copper-doping of phosphates [140,242] and aluminum-doping of LiCoO_2 [189]. With that caveat, the effects of dopant additions on the performance of cathode materials are discussed below.

Although iron-based oxides are not typically used as cathode materials, iron is used as a dopant in nickel-, manganese-, and cobalt-based cathode materials. Iron has been shown to improve the capacity of LiNiO_2 [243,244] and Li_2MnO_3 [245,246], but the beneficial effect diminishes with cycling. Iron doping has also been shown to be beneficial to the performance of $\text{LiNi}_{0.125}\text{Mn}_{0.75}\text{Co}_{0.125}\text{O}_2$ [247], but detrimental to the performance of $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [248]. Similarly, iron impurities have been shown to decrease the capacity and increase the capacity loss during cycling of LiCoO_2 [249]. However, the detrimental effect was attributed to clusters of iron ions, which could be eliminated by annealing the material to disperse the clusters.

Another transition metal that has been used as a dopant for cathode materials is ruthenium, which has been added as a dopant to spinel electrode materials (e.g. LiMn_2O_4 [188] and $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [250]) and to LiFePO_4 [251]. The beneficial effect has been attributed to stabilizing the crystal structure as well as to contributions from the $\text{Ru}^{4+}/\text{Ru}^{5+}$ redox couple. Ruthenium has also been added as RuO_2 to $\text{Li}_3\text{V}_2(\text{PO}_4)_3$ [252] and improved performance by increasing the electrical conductivity.

Chromium forms compounds with the spinel structure and has been added to LiMn_2O_4 [54,83] and $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [239,247,253–255]. Chromium reduces the ordering of lithium ions

in LiMn_2O_4 , which stabilizes the single phase spinel structure [256], and has been shown to increase the capacity retention during cycling for spinel electrode materials, including LiMn_2O_4 [54,83] and $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [254,255]. Nuclear Magnetic Resonance (NMR) analysis indicates the oxidation of Mn^{3+} to Mn^{4+} , and thus the associated lithium deintercalation, is not uniform, but rather occurs preferentially near the chromium dopant ions [257]. Small amounts of chromium additions have also been shown to improve the performance of other cathode materials including $\text{Li}[\text{Mn}_{0.5}\text{Ni}_{0.5}\text{O}_2]$ [258,259], $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [260], V_2O_5 [261] and $\text{Li}_3\text{V}_2(\text{PO}_4)_3$ [262].

Zinc-doping has been shown to improve the performance of $\text{Li}[\text{Mn}_{0.4}\text{Mn}_{0.3}\text{Co}_{0.3}\text{O}_2]$ [263], LiFePO_4 [264], and, through addition of zinc to the electrolyte, LiMn_2O_4 [265] by stabilizing the respective crystal structures. However, the beneficial effect of zinc is usually when added in a coating of ZnO (e.g. $\text{LiNi}_{0.5}\text{Mn}_{0.25}\text{Co}_{0.25}\text{O}_2$ [266], $\text{Li}[\text{Ni}_{0.42}\text{Mn}_{1.42}\text{Co}_{0.16}\text{O}_4]$ [91], $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [267,268], LiFePO_4 [269]), ZnMn_2O_4 (e.g. LiMn_2O_4 [270]) or $\text{Zn}_3(\text{PO}_4)_2$ (e.g. LiCoO_2 [271]) where it reduces reaction between the electrode and electrolyte.

Titanium is added as a dopant in layered structures ($\text{Li}(\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3})\text{O}_2$ [248], $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [272], LiNiO_2 [273]), spinels (LiMn_2O_4 [274–276], $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ [277]) and phosphates (LiFePO_4 [278], LiMnPO_4 [126]). Although not beneficial for LiNiO_2 [273], titanium is beneficial when co-doped with cobalt (i.e. $\text{LiNi}_{0.8}\text{Co}_{0.2-x}\text{Ti}_x\text{O}_2$) [272]. The improved performance is attributed to titanium stabilizing the crystal structure (e.g. $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [272], $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [248]) or reducing dissolution of the electrode [274–276]. Titanium from impurities in the precursor has been shown to occupy iron sites in LiFePO_4 and improve electrode performance [279]. Titanium is also added as a TiO_2 coating to reduce electrode dissolution in the electrolyte [195,280], but degradation of the TiO_2 can lead to degradation in cell performance [180].

Zirconium has similar effects on cathode performance as titanium. Zirconium doping has been used to stabilize the layered crystal structure (e.g. LiCoO_2 [23] and $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [281]) or in LiFePO_4 [282–284] to increase the lattice parameter. Zirconium has also been added as a ZrO_2 or $\text{Zr}(\text{OBU})_4$ coating to reduce reaction of the electrolyte with cathode materials with layered (e.g. $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [177,210] and $\text{Li}[\text{Li}_{1/6}\text{Ni}_{1/6}\text{Mn}_{1/2}\text{Co}_{1/6}\text{O}_2]$ [285]) and spinel (LiMn_2O_4 [183,286] and $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [287]) structures.

Aluminum is a very commonly used dopant in cathode materials. In some cases small amounts of aluminum doping improve the capacity of electrode materials (e.g. LiCoO_2 [189,288], $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ [289–291] and LiFePO_4 [138]), but in most cases the capacity is decreased (e.g. LiCoO_2 [189,288], $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ [289,292], $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [248,260,293–295], other $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ compositions [295–297], LiMn_2O_4 [83,291,298]). The decreased capacity is expected since Al^{3+} cannot be further oxidized, so each transition metal ion replaced with aluminum represents one less oxidizable ion. The observed increases have been attributed to improved electrode kinetics, structural modifications and microstructural effects. For example, the addition of aluminum to LiCoO_2 results in an increase in lattice parameter c [288,299], which facilitates lithiation and delithiation. Aluminum doping has also led to improvements in retention of capacity during cycling (e.g. $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ [294,297], LiMn_2O_4 [83,298]), and performance at high discharge currents (e.g. $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ [248,296], LiMn_2O_4 [291]). However, there are also cases where cycling performance is degraded with aluminum additions (e.g. LiCoO_2 [23], LiNiO_2 [273], $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [260,293]). Aluminum is commonly used as a co-dopant with cobalt in LiNiO_2 (i.e. $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$) for improved stability [211,300–303]. Aluminum can also be added as an alumina coating and has been

shown to improve the capacity (e.g. LiCoO_2 [304], $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$ [52,91], $\text{LiMn}_{1.5}\text{Ni}_{0.42}\text{Zn}_{0.08}\text{O}_4$ [267], LiMn_2O_4 [184]), capacity retention during cycling (e.g. LiCoO_2 [304,305], $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [306], $\text{Li}(\text{Mn},\text{Ni},\text{Co})\text{O}_2$ [91,286,307], $\text{LiMn}_{1.5}\text{Ni}_{0.42}\text{Zn}_{0.08}\text{O}_4$ [267], LiMn_2O_4 [286]) and performance at high discharge currents [91,267]. The improvements are attributed to improved charge transfer kinetics and improved stability with the electrolyte. Beneficial effects have also been attributed to an $\text{Al}(\text{OH})_3$ layer, which has led to its use as a coating for LiCoO_2 [308]. Mixed aluminum oxides, including aluminum-cobalt oxide [309], $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG) [310] and $\text{La}_3\text{Al}_5\text{O}_{12}$ (LAG) [311], have also been used to improve retention of capacity during cycling. Other forms of aluminum that have been shown to improve electrode performance and stability include AlF_3 [312–315], AlPO_4 [91,316–318] and $(\text{NH}_4)_3\text{AlF}_6$ [319].

Magnesium doping generally improves the performance of phosphate electrodes, including LiFePO_4 [138,174,320,321] and LiMnPO_4 [126,140]. The beneficial effect is generally attributed to magnesium increasing the lattice parameter, which facilitates delithiation and stabilizes the structure. Magnesium doping has been reported to improve oxide electrodes by modifying the microstructure (e.g. LiCoO_2 [322]), or reducing charge transfer resistance (e.g. $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [323]), but more often has little, or even a detrimental, effect on electrode performance (e.g. LiCoO_2 [23,271], $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ [324], $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ [260]). However, when present in MgO [325] or $\text{Mg}_3(\text{PO}_4)_2$ [271] coatings, magnesium additions improve electrode performance by reducing the reaction of LiCoO_2 with the electrolyte. On the other hand, doping with a larger alkaline-earth cation (e.g. calcium [326]) can lead to an increase in lattice parameter of LiCoO_2 and improved capacity.

Lanthanum additions have been shown to improve the performances of LiFePO_4 [327,328] and LiCoO_2 [329] cathodes. One of the benefits of the lanthanum additions is that with LiCoO_2 a Li-conducting phase ($\text{La}_2\text{Li}_{0.5}\text{Co}_{0.5}\text{O}_4$) is formed, which improves ion transfer across the electrode–electrolyte interface. Such contact resistance is particularly important in solid–solid contacts and oxide additions can also improve contact between cathode particles or between the electrode and electrolyte materials. Some examples include CeO_2 , which has been shown to reduce contact resistance in LiFePO_4 cathodes [330], and Li_2SiO_3 (and to a lesser extent SiO_2), which has been shown to improve the performance of LiCoO_2 with a solid electrolyte [331–333].

As mentioned above, some additions improve performance by reducing reaction with the electrolyte. Other oxides added to improve capacity retention by reducing reaction and/or forming a beneficial reaction product with the electrolyte include Y_2O_3 [334], YPO_4 [305], B_2O_3 [182], SiO_2 [335], Bi_2O_3 [91,267], Sb_2O_3 [336] and SnO_2 [286]. Some of these same elements when added as a dopant have been reported to improve cycling performance by stabilizing the crystal structure. For example, bismuth has been used as a dopant in LiMnO_2 [215] and LiMn_2O_4 [337], while tin has been used as a dopant in LiMn_2O_4 [338]. In such cases there may be multiple benefits as some the oxide may dissolve in the electrode (when an oxide coating is used) or some of the dopant may form a separate phase on the electrode surface (when the cathode is doped). Other dopants that stabilize the crystal structure include rhodium in $\text{LiCo}_{0.3}\text{Ni}_{0.7}\text{O}_2$ [339], copper in LiMnPO_4 [140] or LiMn_2O_4 [215] and indium in LiMnO_2 [340]. The effectiveness of indium doping is enhanced by co-doping with sulfur, which occupies the oxygen site [340].

Another dopant that occupies the anion site is fluorine, which is a common element in lithium ion batteries. Fluorine is present in the commonly used LiPF_6 -based electrolytes [7], as well as in fluorides, such as carbon fluorides, that are used as anodes in lithium ion batteries [315,341]. Although fluoride compounds have been reported as cathode materials (e.g. iron oxyfluoride [342,343]), in cathodes fluorine is more often added to replace oxygen or in a

compound as an additive to oxide cathode materials [315]. For example, fluorine doping has been shown to improve the capacity of spinel cathodes, $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [344] and $\text{LiMn}_{1.8}\text{Li}_{0.1}\text{Ni}_{0.1}\text{O}_4$ [345], by increasing the lattice parameter and decreasing the average manganese valence. Fluoride additions have also been shown to improve the cycling performance of $\text{Li}(\text{Ni},\text{Mn},\text{Co})\text{O}_2$, either as a dopant [346] or a second phase (e.g. LiF [347], SrF_2 [348]) by reducing reaction with the electrolyte.

The electrode reaction involves lithium ions and electrons, so one approach to improving electrode performance is to add a conducting phase to enhance charge transfer. A sputtered gold layer has been used to improve the performance of LiFePO_4 [349], but this involves an expensive material and process. Silver is lower in cost than gold and has been shown to improve the performance of LiCoO_2 [207,350], LiMn_2O_4 [207] and $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [351] cathodes. The beneficial effect of silver is generally attributed to increased conductivity, but increases in lattice parameter have also been reported in LiCoO_2 [350] and $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [351]. Although these metals can improve performance, the most common addition for improving electrode conductivity is carbon.

Carbon is commonly added to cathodes to more effectively utilize the active cathode material, especially at high discharge rates. For example, carbon additions improve the performance of $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [175,176,352], spinels (LiMn_2O_4 [353] and $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [354]), and especially LiFePO_4 [161,355–357], which has a relatively low electrical conductivity. The effectiveness of carbon additions depends on the amount and type of carbon used. A thin carbon layer can provide a path for electrons without blocking access for lithium ions [358,359]. There is an optimal amount of carbon, which depends on the microstructure and operating conditions, but is typically on the order of 2–10 wt% [197,198,360–364]. Graphitic carbon generally provides higher conductivity and thus higher rate capacities at large discharge rates, so carbons with large sp^2/sp^3 ratios are generally preferred [111,199,202,365]. The particle shape is also important, as nano-scale fibers [158,191,366–370] and high surface area forms of carbon, such as acetylene black [192,371], have been shown to be effective in improving cathode performance. In addition to providing paths for electrical conduction, nano-scale fibers can also be used to disperse the cathode particles for increased contact with the electrolyte [372]. The type and morphology of the carbon deposits depends on the source of the carbon [373], which include glucose [374–376], sucrose [156] and malonic acid [377,378], as well as oligomers [379] and polymers [380,381]. In addition to being used as precursors for carbon, polymers can be used in the electrode. In particular, semiconducting [170,382] or conducting polymers (e.g. polypyrrole (PPy) [162,201,383,384] and polyaniline [385,386]) have been used as additives in cathodes.

6. Effect of microstructure and morphology

As mentioned above, electrode performance depends on the electrode microstructure and morphology. Although the focus of this paper is on the materials rather than morphology, some general aspects of electrode morphology will be discussed. Intercalation and deintercalation occur along specific crystallographic planes and directions, so higher crystallinity improves electrode performance (e.g. LiCoO_2 [387], LiMn_2O_4 [388], $\text{Li}_{1.02}\text{Mn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ [389], LiFePO_4 [155,390–392]).

The electrode reaction occurs at the surface and requires transport of ions into the electrode material, so small particles, which provide high surface area [388] and short diffusion distances [393], and are generally desired (e.g. LiCoO_2 [164,394,395], $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ [396], LiMn_2O_4 [181,397,398] and LiFePO_4 [155,157,171,399–402]). However, particles can become too small,

so that processes other than the surface reaction, such as diffusion of ions in the electrolyte to the particle surface, become rate limiting. In addition, the high reactivity of nanosize particles can be disadvantageous in terms of safety and stability during long operational lifetimes [12,403]. Thus, intermediate particle sizes sometimes exhibit the best performance [404–406]. For example, analysis of the discharge of individual LiCoO_2 particles suggests that commercially available micron-size particles have sufficient capacity, so nanoparticles may not be necessary [407]. Similarly, micron-scale carbon fibers have been used to provide a framework for dispersion of LiFePO_4 particles to improve access to lithium ions in the electrolyte [408]. In addition, control of porosity is important to allow for access of the electrolyte to the electrode surface [194,409–412]. For example, a recent report has shown that nanoporous micron-size particles perform better than nanoparticles [413].

Particle shape is important for electrode performance. For LiMn_2O_4 spinel electrodes, spherical particles have been reported to provide the best performance [414,415], which may be related to the more isotropic lithium transport in the spinel structure, as compared to layered structures. For layered structures, including LiCoO_2 [416] and $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ [417], however, non-spherical particles have been shown to improve performance, which has been attributed to high tap density. Rod- or needle-shaped particles, which have large surface-to-volume ratios, have also shown good performance, especially during cycling and at high discharge currents [418–420]. Rod-shaped particles can also be useful as precursors to produce small nano-scale spherical particles [421]. Plate-like particles, which reflect the layered crystal structure, have also been used [204,422,423], and can better accommodate expansion/contraction during intercalation/deintercalation and reduce cracking. In addition to the overall particle shape, the surface morphology is also important. For example, a “desert rose” surface morphology on LiCoO_2 produces a high surface area, which results in a high capacity [424].

The production of small power supplies for miniature devices requires thin-film based batteries, so thin-film deposition of cathode materials is required for these applications [425]. Thin-film deposition techniques, such as pulsed laser deposition and sputtering, have been used to produce cathode materials including LiCoO_2 [426–430], LiMn_2O_4 [431–435], LiFePO_4 [436]. The small batteries have high energy densities, but the energy density can be further improved by using three-dimensional architectures [437,438]. Such three-dimensional geometries require templates for control of the size, shape and arrangement of the battery components [439]. Approaches for producing such complex geometries can be inspired by biological systems [440], and can even use biological processes for fabrication [441–443]. The processes required to fabricate these complex geometries will likely place additional constraints on materials selection, so further developments in cathode materials and fabrication processes will be required.

7. Conclusions

The development of improved cathode materials is a challenge for meeting current and future energy storage requirements. Several transition metal based cathode materials can provide high voltages and good capacities. Full utilization of these materials for numerous recharging cycles and at high discharge currents continues to be a challenge. Specifically, stabilizing the desired crystal structure, especially during delithiation, and preventing reaction with the electrolyte are important for long operational life, while improved transport to and in the electrode are important for achieving high discharge current. Progress has been made by

engineering the electrode composition, microstructure and morphology, but additional improvements are needed.

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