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A versatile single molecular precursor for the synthesis of layered oxide cathode materials for Li-ion batteries

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A carbonyl-bridged single molecular precursor $LiTM(acac)_3$ (TM = Ni/Co/Mn, acac=acetylacetone) featured 1D chain structure, was designed and applied to achieve the layered oxide cathode materials $LiTMO_2$ (TM=Ni/Co/Mn, NMC) here. As representatives, layered oxides, primary $LiCoO_2$, binary $LiNi_{0.8}Co_{0.2}O_2$ and ternary $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ were successfully prepared as the cathode materials. When they are applied to Li-ion batteries (LIBs), all exhibit good electrochemical performance due to unique morphology and great uniformity of element distribution. This versatile precursor is predicted to accommodate many other metal cations, such as Na^+, Al^{3+}, Fe^{2+}, due to the flexibility of organic ligand, which not only facilitates the doping-modification of NMC system, but also enables synthesizing Na-ion layered oxides. It opens a new avenue to synthesize high-performance layered oxide cathode materials for LIBs.

Layered oxides cathode materials, including $LiCoO_2$ and its series derivatives Li(Ni, Co, Mn)O_2 (NMC), as the main options for the cathode materials of Li-ion battery, has implemented industrial production because of their good comprehensive performance, including large capacity, good cycling and rate capability.[1-6] Thereinto, $LiCoO_2$ is the first commercialized cathode material, and still shares the biggest market due to the high volumetric energy-density and easy preparation.[7] However, it also presents some drawbacks, such as relatively low practical capacity (around 140 mA h g^{-1}), high cost due to the rarity of Co source, and high toxicity of Co compared with Ni, Mn and Fe.[8, 9] So developing new cathode materials with high comprehensive performance attracted a lot of attention and efforts. Doping and replacement of Co with other TM elements becomes one of the successful strategies to tailor and enhance the electrochemical performance of cathode materials. Based on $LiCoO_2$, ternary Ni-Co-Mn system with different TM ratios, has been developed. Thereinto, Ni substitution is to improve the specific capacity with low cost, and Mn substitution is to enhance the structural stability of layered oxide.[10-12] So, multiple TM substitution provides a vast potential to enhance the comprehensive electrochemical performance of layered materials.

With the advantage of tailoring properties by multiple TM substitution, it brings with another difficulty to implement uniform distribution of multiple metal elements in such a complicated polynary layered oxides system. In the traditional solid state method, mixing Li source and multiple TM sources by grinding or ball milling could not implement the great uniformity in precursors on nanometer or atomic scale. Thereinto, it is much more difficult to achieve the uniform distribution of TM elements with Li due to their much lower thermal diffusivities than Li.[13, 14] To solve this problem, a co-precipitation method was developed. It combines multiple TM elements into one compound, such as TM hydroxide,[15-17] TM carbonate,[18-20] or TM oxalates,[21] to implement the uniformity of TM elemental distribution, and then to mix it with Li-source to get layered oxides through high temperature calcination.

Inspired by the co-precipitation method, the best strategy to implement the uniform distribution of all metal elements is to combine all the involving metal elements (including Li and TM) in one single compound with correct ratios. This method is called as single molecular precursor method. Taking layered oxides $LiTMO_2$ as an example, it requires an appropriate ligand to combine Li and TM into one compound with Li/TM ratio as 1. Till now, there are only five cases to report the synthesis of cathode materials through single molecular precursor method by Dikarev group and Schneider group. Two of them are the synthesis of layered $LiCoO_2$.[22, 23] The other three are the synthesis of spinel $LiMn_2O_4$ and layered $LiFeO_2$.[24-26] As we know, the synthesis of polynary layered oxides involving multiple TM cations, and their electrochemical performance through single molecular precursor method has never been demonstrated before. In addition, various organic ligands provide more choices than the limited inorganic precipitant (such as OH^- and Co_3^{2-}), which might be beneficial to the co-precipitation efficiency of multiple metal cations.[27, 28]

In this work, a carbonyl-bridged single molecular precursor $LiTM(acac)_3$ (TM = Ni/Co/Mn, acac=acetylacetone) featured 1D chain structure, was designed and applied to achieve the layered oxide cathode materials $LiTMO_2$ (TM=Ni/Co/Mn) here. Acac as carbonyl-rich ligand was chose to implement precise control of metal element compositions in single molecular precursors to lead to the correct compositions and great uniformity in the final products. As representatives, layered oxides, $LiCoO_2$, $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$

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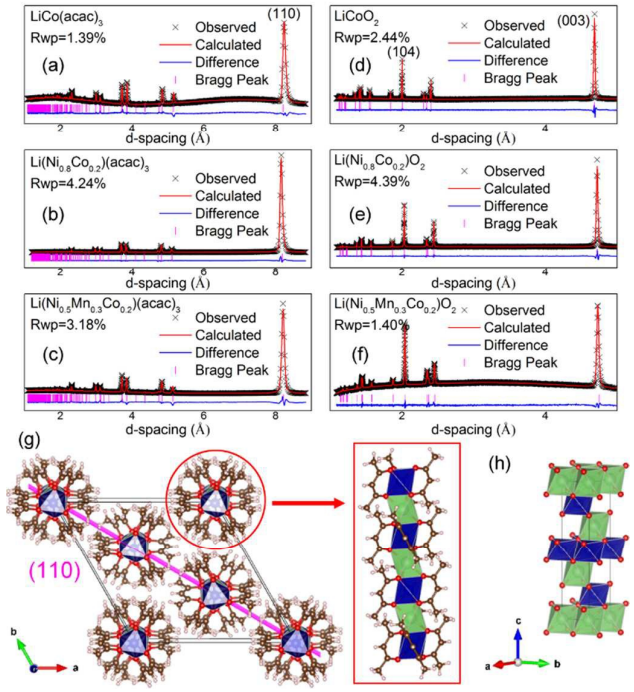

Fig. 1 Experimental XRD patterns of $LiCo(acac)_3$ (a), $Li(Ni_{0.8}Co_{0.2})(acac)_3$ (b) and $Li(Ni_{0.5}Mn_{0.3}Co_{0.2})(acac)_3$ (c); experimental XRD patterns of $LiCoO_2$ (d), $Li(Ni_{0.8}Co_{0.2})O_2$ (e) and $Li(Ni_{0.5}Mn_{0.3}Co_{0.2})O_2$ (f); representative structural sketch maps of $LiCo(acac)_3$ (g) and $LiCoO_2$ (h).

were successfully prepared through their single molecular precursors ($LiCo(acac)_3$, $LiNi_{0.8}Co_{0.2}(acac)_3$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}(acac)_3$), respectively. They all exhibited good electrochemical performance due to the unique morphology and the great uniformity of elemental distribution.

The single molecular precursors were synthesized by the solution reflux method (details in ESI). As shown in Fig. S2, $LiCo(acac)_3$, $Li(Ni_{0.8}Co_{0.2})(acac)_3$ and $Li(Ni_{0.5}Mn_{0.3}Co_{0.2})(acac)_3$ presented pink, lavender and light grey colors, respectively, with different TM compositions. The powder X-ray diffraction (XRD) pattern of $LiCo(acac)_3$ was illustrated in Fig. 1a, which was consistent with the previous report.[1] The patterns of $LiNi_{0.8}Co_{0.2}(acac)_3$ and $LiNi_{0.5}Mn_{0.3}Co0_{.2}(acac)_3$ (Fig. 1b-c) were similar with that of $LiCo(acac)_3$, which indicated that they all crystallized in a same space group (*R*-3*c*). The ICP-AES results (Table S1) indicated that the elemental compositions of Li and transition metals (TMs) were precisely controlled in the individual compound as designed. About 1% Li loss was observed after calcination. Their crystal structure was illustrated in Figure 1g. As we can see, it was constructed by a lot of parallel-aligned 1D chains along the c axis, which were consisted by the alternately-connected LiO_6 and TMO_6 octahedra within a face-sharing mode. Thereinto, each TMO_6 octahedron was composed by a TM cation chelated with three acac molecules, and one LiO_6 octahedron was composed by a Li ion bridged by six acac molecules. As shown in Figure 1h, the LiO_6 and TMO_6 octahedra are the basic structural units to produce the layered oxides $LiTMO_2$.

To study the effect of TM compositions on the crystal structure, Rietveld refinements were performed for the three XRD patterns by using the reported $LiCo(acac)_3$ structure as the initial model structure, and the relevant crystallographic parameters were deposited in Table S2 and Figure S2. As shown in Figure S2a, the unit cell parameters, a and c, firstly decreased with 80% Co(II) substitution with Ni (II), then increased with further 30% Ni(II) substitution with Mn (II). On the other hand, this unique structure reflected that the organic ligand (acac) presented a much higher flexibility than the inorganic anions, OH^- and CO_3^{2-}, which could accommodate a much larger variation of metal-O bond lengths and long angles by the deformation of organic ligands. So it is the in-depth reason why the organic ligand could combine Li ions and various TM cations into one crystal framework. This character also allowed other metal cations to enter into the framework, thereby provide a great potential for various elemental doping.

The corresponding layered oxides $LiCoO_2$, $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ were prepared by calcining the corresponding precursors under preliminarily optimized conditions. Their structures were measured by powder X-ray diffraction (Fig. 1d-f). All diffraction peaks can be indexed to hexagonal $LiCoO_2$ (*R*-3*m*) with a layered structure (JCPDS no. 75-0532), revealing high phase purity for them. The (003) and (104) peaks of $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ shifted to high d-spacing direction than $LiCoO_2$, indicating larger lattice parameters after Ni and Mn substitution. Integrated intensity ratio of (003) to (104) peaks (denoted as I (003)/I(104)) was usually adopted to roughly evaluate the cationic disordering in layered structure.[2] Herein, high I(003)/I(104) values for $LiCoO_2$ and $LiNi_{0.8}Co_{0.2}O_2$ (1.35, and 1.22, respectively), implied much less Li/Ni disordering in their layered structure, which hinted good electrochemical performance for them. Rietveld refinements were also performed for these three XRD patterns, and the relevant crystallographic parameters were deposited in Table S3 and Figure S2. As shown in Figure S2b, the lattice parameters, a and c, increased with the Ni and Mn substitution, which is consistent with the previous report.[12] The occupancy of Ni at 3b site (denoted as Ni(3b)) was another important indicator to show the Li/Ni disordering. Now the low Ni(3b) value (0.0394) in Table S3 also predicted good electrochemical performance for $LiNi_{0.8}Co_{0.2}O_2$.

The morphology of precursors and corresponding layered oxides were investigated by SEM characterization. As shown in Fig. 2, all the precursors presented prism-like shape, with length about 10-20 μm and width about 2-4 μm. This morphology was consistent with their hexagonal crystal structure shown in Fig. 1g, because the crystals preferred to grow up along the c axis by extending the 1D chain. The layered $LiCoO_2$ could be obtained by very low temperature, 450 ºC (Fig. S6) with particle size around 200 nm (Fig. S7). When calcined at higher temperature (850 ºC), the nanocrystals merged and grew significantly by about ten times. As shown in Fig. 3a, a thick plate was composed by small particles with size around 2 μm. The high-magnification SEM images in Fig. 3b showed the micrometer pillar shape with surface-exposed layered texture, a unique morphology for $LiCoO_2$ that has never been reported before. These surface-exposed layered textures could be assigned to (003) crystal plane according to its layered structure shown in Fig. 1h, which might be beneficial to Li^+ fast de/intercalation to enhance the rate capability. After sintering $LiNi_{0.8}Co_{0.2}(acac)_3$ at 800 ºC for 8 h, $LiNi_{0.8}Co_{0.2}CoO_2$ presented a 3D network framework composed with small particles (Fig. 3c) with the size about 500 nm (Fig. 3d). As shown in Fig. 3e-f, $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ showed a similar 3D network morphology constructed with much smaller particles. The EDX elemental mappings were performed for three materials to study the elemental distribution of transition metals. Fig. S9 displayed that Ni, Mn and Co distributed uniformly in these samples. In addition, the chemical compositions of three single molecular precursors and the corresponding layered oxides were measured by ICP-AES

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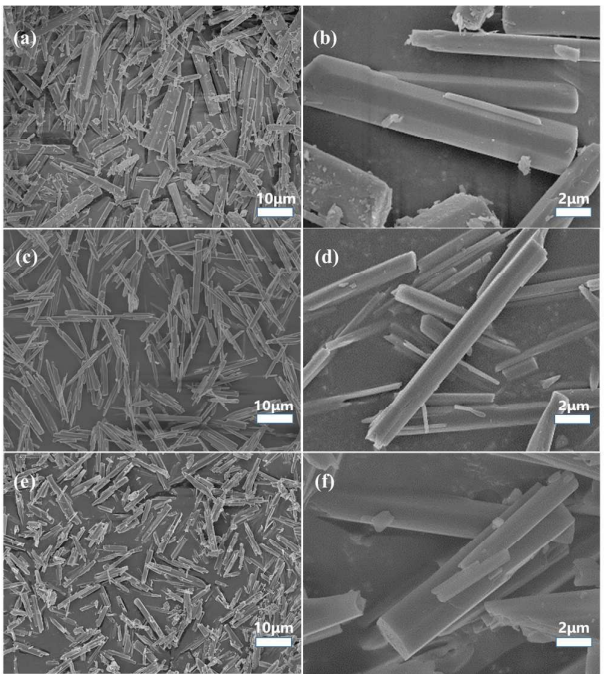

Fig. 2 Low magnification (a) and high magnification (b) SEM images of $LiCo(acac)_3$; Low magnification (c) and High magnification (d) SEM images of $LiNi_{0.8}Co_{0.2}(acac)_3$; Low magnification (e) and High magnification (f) SEM images of $LiNi_{0.5}Mn_{0.3}Co_{0.2}(acac)_3$.

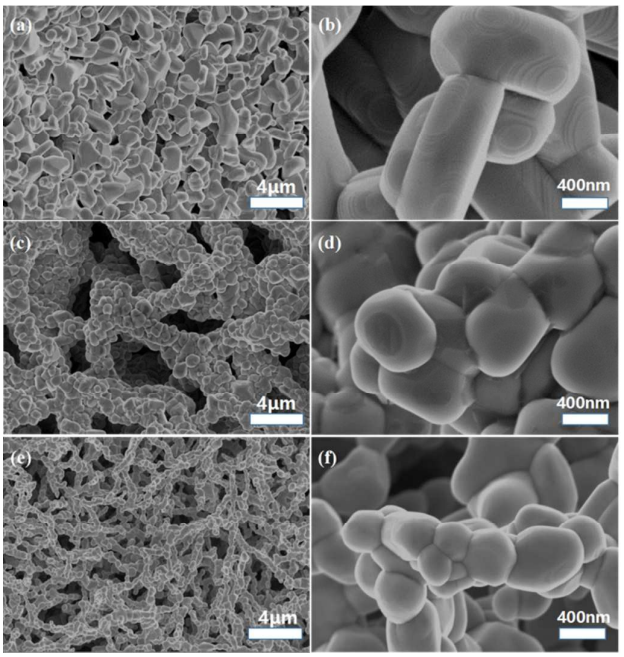

Fig. 3 Low magnification (a) and high magnification (b) SEM images of $LiCoO_2$; Low magnification (c) and High magnification (d) SEM images of $LiNi_{0.8}Co_{0.2}O_2$; Low magnification (e) and High magnification (f) SEM images of $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$.

(Table S1). It is clear that, the molar ratios of metal elements including Li, Ni, Co, and Mn were consistent with the pre-designed. After calcination, the molar ratios of metal elements nearly kept constant. It further proved that the elemental composition could be precisely controlled by this single molecular precursor method.

Finally, $LiCoO_2$, $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ were assembled into half cells to test their electrode performance (see Experimental Section in the ESI†). Fig. 4a showed the charge and discharge profiles of the first two cycles with a current density of 18 mA g^{-1} between 2.7 and 4.4V for $LiCoO_2$. 1st charge and discharge capacities were 180.9 and 171.3 mA h g^{-1}, and the capacities for 2nd cycle were 170.5 and 170.1 mA h g^{-1}, respectively. The coulombic efficiencies for the 1st and 2nd cycle were calculated to be 94.6% and 99.7%, respectively. So large capacities were superior to most of $LiCoO_2$ samples prepared by traditional solid state method and other methods.[29-32] The small capacity loss during the 1st cycle could be ascribed to the irreversible loss of Li ions caused by the formation of SEI films. Fig. 4b exhibits the electrode capacities tested at the current densities increasing from 18 to 36, 90, 180, 360, 900, and 1800 mA g^{-1}, and five cycles were performed at each rate. The corresponding discharge capacities could reach 170, 165, 160,155, 146, 128, and 86 mA h g^{-1}, respectively. The reversible capacity recovered to 163 mA h g^{-1} when the current density was brought back to 18 mA g^{-1}. Fig. 4c further illustrates the cycling performance of $LiCoO_2$ with a current density of 180 mA g^{-1} at 25 ºC, which can retain a reversible capacity of 136.6 mA h g^{-1} with capacity retention of 88.7% after 50 cycles.

Similarly, Fig. 4d sowed the charge and discharge profile of $LiNi_{0.8}Co_{0.2}O_2$ with a current density of 18 mA g^{-1}. It presented the charge and discharge capacities of 193.8 and 181.13 mA h g^{-1} for the 1st cycle, 183.4 and 180.9 mA h g^{-1} for the 2nd cycle. Higher capacities and lower voltage plateaus was observed by comparing it with pure $LiCoO_2$ after Ni substitution, which was consistent with previous reports.[33-35] The coulombic efficiencies were also very high, 93.4% and 98.6% for the 1st and 2nd cycle, respectively. Fig. 4e presented an excellent rate capability, which was similar with pure $LiCoO_2$. Fig. 4f further illustrates the cycling performance of $LiNi_{0.8}Co_{0.2}O_2$. The capacity retention was 81.5% after 50 cycles, which was lower than pure $LiCoO_2$. It might be due to high Ni reactivity and structural reconstruction on the particle surface for high Ni layered oxides.[36] These excellent cycling and rate performance could compare to or superior to that of $LiNi_{0.8}Co_{0.2}O_2$ prepared by traditional solid state method and other methods.[37-39] Fig. 4g-i showed the charge and discharge profiles, rate and cycling performance for $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, respectively. It was clear that the charge/discharge capacities decreased in comparison with $LiCoO_2$ and $LiNi_{0.8}Co_{0.2}O_2$ after Mn substitution. The rate performance was also deteriorated after Mn substitution. Nevertheless, its cycling performance shown in Fig. 4i was greatly increased due to Mn replacement. A super high capacity retention of 98.5% was obtained after 50 cycles. In one word, Mn substitution decreased the capacity and rate capability, but increased the cycling stability. This result is in accord with Mn role in layered oxide, i.e., improving the structural stability.[11] In addition, the low capacity and poor rate capability might be due to high Li/Ni mixing and small grain size shown in Table S3.

In summary, multiple layered oxides $LiTMO_2$, including $LiCoO_2$, $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, were prepared by the thermal decomposition of a versatile precursor $LiTM(acac)_3$. They not only present unique morphology with great uniformity of element distribution, but also exhibit good electrochemical performance. It provides a unique and valuable approach to prepare high performance electrode materials for LIBs. More layered cathode materials, such as NCA, NCM with other TM ratios, even including layered sodium ion materials, are expected to be prepared by this method.

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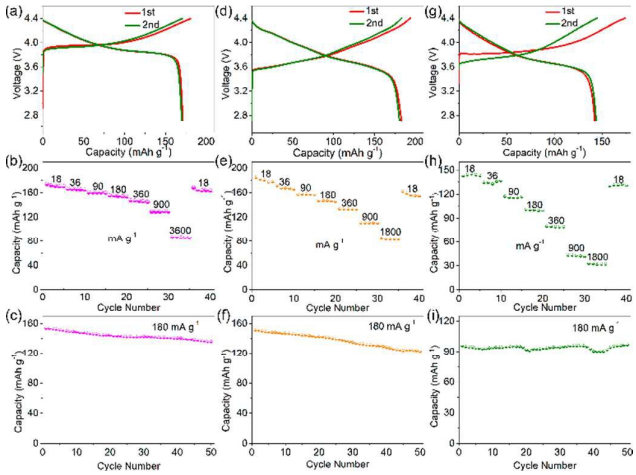

Fig. 4 Electrochemical performance of $LiCoO_2$, $LiNi_{0.8}Co_{0.2}O_2$ and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$. Charge and discharge profile at selected cycles with a current density of 18 mA g^{-1} for $LiCoO_2$ (a), $LiNi_{0.8}Co_{0.2}O_2$ (d) and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ (g); rate performance at various current densities for for $LiCoO_2$ (b), $LiNi_{0.8}Co_{0.2}O_2$ (e) and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ (h); and cycling performance with a current density of 180 mA g^{-1} at 25□ for for $LiCoO_2$ (c), $LiNi_{0.8}Co_{0.2}O_2$ (f) and $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$ (i).

Conflicts of interest

There are no conflicts to declare.

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