A review of MOFs and its derivatives for lithium ion battery anodes

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Abstract. With the development of electric vehicles and clean energy, the demand for lithium-ion batteries (LIBs) has increased significantly in recent decades. As traditional anode materials can only basically meet the requirements of current consumer electronics products, metal-organic framework (MOFs) material and its derivatives attract great attention to become a potential substitute with advanced performance. Pristine MOFs material has greater development potential in terms of high energy density anode. MOFs-derived materials, including porous carbon material, metal oxide and composite material, could exhibit improved electrochemical behaviours and better stability. This article introduces the pristine MOFs and MOFs-derived materials as an anode in lithium-ion batteries, as well as their modification methods that have been widely studied at the current stage. In the end, we discussed the future development trends of various MOFs materials.

1. Introduction
Lithium-ion battery (LIB) is a very promising energy storage system, which is widely used in phones, computers, and other portable electronic devices. It is also an important power supply source for electric vehicles and emerging smart grids in the future. Anode, an indispensable part in the battery system, is responsible for energy storage by storing as many lithium ions as possible. However, the theoretical specific capacity of today's commercial graphite anode is only 372 mAh/g, which cannot satisfy the requirements of the next generation of high-energy LIBs [1]. Thus, it is urgent to develop new anode active materials with higher energy density, good rate performance and low price.

The concept of MOFs (Metal–Organic Frameworks) was first proposed by Yaghi [2] in 1995, which is defined as a porous material with a periodic network formed by self-assembly of inorganic metal centres and bridged organic ligands [3]. Different from traditional graphite material and alloy material, it has the remarkable characteristics of high specific surface area, high porosity, good thermal stability and ordered crystal structure, etc. As a kind of material receiving tremendous attention, it has been widely used in gas storage and separation, catalysis, sensing, luminescence, drug carrier [4]. Recently, MOFs and its derivatives have been gradually applied in energy storage, and they are considered potential anode materials for high-performance LIBs due to their high specific capacity and high cycle life.

This article introduces pristine MOFs anode, MOFs-derived anode, and their common modification methods that have been widely studied at the current stage.
2. Pristine MOFs material

Unlike graphite, a commercial LIBs anode material with a layered structure, the pristine MOFs material has a diverse structural framework composed of metal centres and organic ligands bridged with them. Therefore, it exhibits an adjustable porous structure and a large specific surface area that can provide more electrochemical active sites. There are two main methods for pristine MOFs materials to store lithium ions: intercalation/de-intercalation and conversion reaction mechanisms [5].

Based on the regular and rich pore structure of MOFs, a large number of lithium ions can be quickly transferred and intercalate or deintercalated into the pores without causing structural changes of the frame, which is reversible intercalation/de-intercalation. Thus, MOFs shows the potential of large reversible capacity and good rate performance. As for the conversion reaction mechanism, it refers to the conversion of valence. As we know, redox reactions will occur during the charging or discharging cycles to transfer charges. In this case, metal ions or metal clusters in MOFs can be used as redox active substances in the redox reaction process. Generally speaking, the larger the valence range of the material is, the more electrons can be transferred, this is one of the reasons why MOFs could have a higher theoretical specific capacity than graphite [6]. Moreover, the metal elementary substance generated after the redox reaction can further store lithium ions through alloying reaction and could further increase the specific capacity.

In 2006, LI and his collaborators [7] first attempted to prepare Zn₄O(1,3,5-benzenetribenzoate)₂ (named as MOF-177) by solvothermal synthesis method and used it as the anode of LIBs. Although the specific capacity of the material is low and the cycle stability is poor, it provided infinite possibilities for MOFs to be used as LIBs anode material. As expect, the following decades witness the dramatic development of pristine MOFs. Until now, it is more than 20,000 MOFs have been discovered, and more and more of them were proved an outstanding performance as anode material [8]. Although the MOFs material has advantages in pore structure and high specific capacity compared with the traditional anode electrode material, its inherent low conductivity and high irreversible capacity loss make it face great challenges in the application of high-performance anode active materials.

For the shortages of the pristine MOFs, various effective modification methods have been developed. Among them, selection of suitable ligands, bifunctionalization [9], element doping, and compounding with conductive materials are favoured by researchers.

For MOFs materials containing unsaturated bonds in their organic ligands, choosing a suitable ligand can contribute to increasing the specific capacity through the reversible lithium ion storage process of opening the unsaturated bonds and then reforming bonds in the charge-discharge process. For example, in 2016, Cheng et al. [10] chose aromatic ligand to synthesize [Co₁.₅L(H₂O)₄]ₙₙ with a one-dimensional chain structure and used it as anode active material for LIBs. After 50 cycles at 50 mA/g, the reversible specific capacity was 431 mAh/g, and the corresponding coulomb efficiency remained constant at 98.3%. Through further research on the electrochemical redox reaction, a mechanism that both metal centers and organic ligands could simultaneously participate in lithium storage was discovered for their high specific capacity. Similarly, Teng et al. [11] synthesized four different pillared-layer structure of MOFs with rich Li-binding sites, the three MOFs materials exhibited high specific capacities (about 600 mAh/g at 100 mA/g) with remarkable rate and cycling performance. It was found that these MOFs materials allowed a high Li insertion only by its aromatic carboxylate ligands and pillared-layer structure, which suggested metal centers were not involved in the lithiation process and had no significant influence on lithium ion storage for those MOFs materials.

In addition, in complex working conditions like the electrochemical environment of LIBs, bifunctional MOFs materials (BMOF) are favored to be synthesized for its better stability and performance. Usually, the rich inner pores of MOFs can be functionalized by chemical grafting and its modified frameworks could be achieved by the post-synthetic method. In 2015, Chen Liang and his collaborators [12] designed and then synthesized a new BMOF, Zn(IM)₁.₅(abIM)₀.₅ with the hydrophobic and polar functionalities. The as-synthesized BMOF showed remarkable thermal and chemical stability in the experiment test, and it performed a reversible Li storage capacity of 190mAh/g at a current density of 100mA/g after cycling for 200 times (Fig. 1).
Figure 1. a) Specific capacity and coulombic efficiency of BMOF electrodes in different current densities. b) Rate cycling performance of BMOF electrode.

Although more and more pristine MOFs with different topology structures or compositions are reported as anode material for LIBs, there are few reports on the improvement of the conductivity of the material itself. Therefore, based on considering the specific capacity and cycling performance, exploring the topology structure of pristine MOFs with faster charge transfer will be one of the future research directions.

### 3. MOFs derived materials

MOFs-derived materials refer to a type of porous nanostructured materials generated by heat treatment or chemical treatment using MOFs as sacrificial templates [13]. The derived material can, to some extent, inherit the characteristics of MOFs themselves, such as high specific surface area, large pore volume and special framework structure. Because of these, MOFs-derived materials have special properties and novel functions in many fields. Till now, lots of MOF-derived anode materials have been reported, involving porous carbon material, metal oxide, and their composites. All of them could demonstrated impressive electrochemical behaviours in LIBs.

#### 3.1. Porous carbon material

Various traditional carbon materials have been successfully synthesized and used in anode account to their abundant reserves and good chemical stability. However, because the energy density and power density of them are difficult to improve, the electrochemical performance is not good enough for future demand. In recent years, porous carbon materials derived from MOFs have broken this embarrassing situation. MOFs derived carbon materials can be pyrolyzed at high temperatures in an inert atmosphere, the carbon materials generated will be endowed with nano-cavity and the open access for small molecules [14]. To a large extent, MOFs-derived porous carbon materials could maintain the characteristics of the precursor high specific surface area, uniform pore size distribution and rich active sites. These advantages make MOFs-derived carbon material a potential to reach larger lithium-ion storage and faster charge transfer.

With the deepening of the study, the researchers found that the porous carbon materials derived from MOFs by heat treatment also have some shortcomings and limitations. This is mainly due to the poor thermal stability of MOFs itself. Even if the heat treatment process is controlled at low temperature, the MOFs-derived carbon material will still collapse to a certain extent [15]. These collapses will impede the insert of lithium-ion in the process of charging and result in a poor capacity.

Therefore, to obtain carbon materials with as much MOFs morphology as possible, element doping has become one of the most effective means to enhance the stability of its skeleton. In the preparation of MOFs-derived porous carbon, heteroatoms such as boron, nitrogen and sulfur could be doped into the carbon skeleton by selecting appropriate organic ligands, which greatly affects its physical properties. Among them, nitrogen doping is most famous. On the one hand, the introduction of nitrogen can increase the charge density of the material, which promotes the transfer of charge and ion, and enhances the
thermal stability of the structure [16]. On the other hand, nitrogen will cause the product with plenty of
nanopores, providing more space for the storage of lithium ions. Zheng [17] reported the nitrogen-doped
graphene particle analogues obtained by direct pyrolysis of a nitrogen-containing MOFs under a
nitrogen atmosphere. Used as electrode material in lithium-ion batteries, these particles had a specific
capacity of 2132 mAh/g after 50 cycles at 100 mA/g (Fig. 2). This excellent electrochemical
performance is attributed to it doped with nitrogen within the hexagonal lattice and edges resulting high
nitrogen content of 17.72% and large specific surface area of 634.6m²/g.

Figure 2. a) Cycling performance and corresponding Coulombic efficiency at 100 mA/g. b) Rate
performance at various currents from 100 mA/g to 1600 mA/g.

It is worth mentioning that hybrid-pores, a method achieved by adjusting treatment parameters, has
also affected the performance of lithium-ion batteries. Chang et al. [18] obtained MOF-525 crystals with
different particle sizes by adjusting the concentration of the regulator, and then annealed at 800 degrees
Celsius to obtain a series of nanoporous carbon with different ratio of micropores to mesopores. By
voltammetric cycle test, the nanoporous carbon materials with particle size of 185nm with optimized
ratio of micropores to mesopores showed the best electrochemical performance. The remarkable
performance resulted from the hierarchical micro/mesoporous structure reducing the ion diffusion
resistance and increasing the contact surface area, which has a positive effect on the lithium storage.
Coincidentally, by the vacuum carbonization of a zinc-based MOFs at 1000 degrees Celsius, Li et al.[19]
synthesized a highly porous pure carbon material consisting of macropores, mesopores, subnanopores
and closed pores, which has an amazing lithium storage capacity of 2458 mAh/g at the current of 74
mA/g and a favourable high-rate performance.

Porous carbon materials by using MOFs as a sacrifice template have promising performance as anode
material in LIBs, but improper carbonization temperature will affect the graphitization degree centigrade
of carbon components and then affect the electrical conductivity of the material obtained. The key
problem of accurately controlling the carbonization process is bound to attract much more researchers'
attention in the future.

3.2. MOFs-derived metal oxides
Transition metal oxides, Fe₂O₃, Co₃O₄, CuO, TiO₂, etc, which can store lithium ions through the
conversion reaction mechanism, has been reported as environmentally friendly electrode materials with
a high theoretical specific capacity. However, it encountered a bottleneck in practical applications.
During the charging or discharging process, the transition metal oxide undergoes a huge volume change
and serious particle aggregation. These cause severe powdering and cracking of the electrode, resulting
in rapid capacity degradation and poor rate performance. With the use of MOFs as sacrificial templates
in calcination, a new type of metal oxides with controlled structure and composition could be obtained
to solve this problem. Compared with other non-porous metal oxides, MOFs-derived metal oxides can
retain the structural characteristics of the precursor. Thereby, they have the following advantages as
electrode material: 1. Controllable particle size and morphology 2. High porosity and larger active
surface improve electrolyte penetration and further improve the utilization rate of active site [20]. 3. The
continuous pore structure shortens the diffusion distance of ions and charges, which is beneficial to molecular dynamics to achieve a better rate performance.

In order to further strengthen the advantages of MOFs-derived metal oxides as an anode, several modification strategies have been developed, including bimetallic oxides, building smart structures, nitrogen element doping and so on.

Among them, bimetallic oxide refers to two kinds of metal oxides of different metal cations, which is different from the physical mixture of two metal oxides. The synergistic effect of the two active metals reduces the activation energy of electron transfer to achieve the purpose of improving conductivity. Besides, the bimetal oxide can also shorten the electron transmission path and ensure the stability of the electrode material under high current [21]. According to reports, Wang and co-workers [22] prepared Co/Ni-MOFs nanorod with uniform distribution and high structural integrity via a one-step facile microwave-assisted solvothermal method, and then obtained Co-Ni-O bimetallic oxide through heat treatment. The initial discharge-charge capacity of this material were 1737 mAh/g and 1189 mAh/g, respectively. At a low current of 100 mA/g, it delivered a reversible capacity of 1410 mAh/g after 200 repetitive cycles, which was significantly larger than the theoretical capacity of corresponding metal oxide. (Theoretical capacities of Co3O4 and NiO are 890 mAh/g and 718 mAh/g, respectively) Moreover, the porous ZnO/ZnCo2O4 nanosheets obtained by Xu et al. [23], which using Zn-Co-MOF as a precursor in one step of annealing, also proved the advantages of bimetal oxide. By using it as anode in LIBs, a reversible capacity of 1016 mAh/g was maintained after 250 cycles at 2A/g, even at a high current density of 10A/g, the capacity could attain over 600 mAh/g (Fig. 3). These outstanding performances have been mainly attributed to the synergistic effect that makes the electrode materials have higher conductivity and better electrochemical reactivity.

Building a smart structure is another common strategy. When converting MOFs into metallic oxides, it is often accompanied by a large volume shrinkage, which leads to the appearance of internal cavities. Meanwhile, the gas generated during heat treatment will cause pores in the shell, resulting in porosity increasing. Reasonable application of this feature can construct a hollow porous or special complex structure, helping to reduce the loss of irreversible capacity. Guo et al. [24] used Co[Fe(CN)6]0.667 as a precursor in calcinate and successfully fabricated CoFe2O4 nanocubes with a hollow porous structure. As an anode material, the specific capacity could be retained at 1115 mAh/g after 200 cycles, and it also performed good rate performance. It is because the hollow structure promoted the uniform distribution of stress and improved the adaptability of the volume change during lithium insertion/extraction. Also, the pores of the outer shell provided extra charge storage sites for redox reactions and enhanced ion diffusion at the same time, so that the fabricated material exhibited excellent cycling stability. Compared with the simple hollow structure, the complex structure can maximize the use of space. One example is MOF-derived Co3O4 with twin hemispherical and flower-like structures, which was obtained by Zhang and co-workers [25]. Tested as an anode in LIBs, this active material has an initial discharge capacity up to 1325.5 mAh/g, and it can still arrive a reversible capacity of 470 mAh/g after 90 cycles. From
authors’ point, it possesses favourable features, such as a special complex structure for fast charge transfer, smaller particle size and compact porous structures for promoting the electrolyte to enter the electrode material.

At present, there are more and more research reports on the porous, hollow and complex structures of MOF-derived metal oxides. In addition to enjoying the application advantages brought by the structural complexity, exploring simple and facile synthesis methods is also one of the potential research directions.

3.3. Metal oxide / carbon composite materials

Based on the inherent structure of MOFs, it is both a metal source and a carbon source. In the inert gas atmosphere, the organic ligand components after calcination of MOFs usually decompose to form Co2, NOx and other gases, and carbon will be generated when oxygen is insufficient [26]. Therefore, suitable control of heating parameters can obtain two components at the same time, namely metal oxide/carbon composite materials. This method makes full use of the particularity of the MOFs skeleton, so that the metal oxide can be uniformly dispersed in the porous nano-carbon matrix. As a result, it not only solves the problem of difficulty in controlling particle size in traditional synthesis, but also effectively alleviates the volume expansion of metal oxides during cycling and improves electrical conductivity to a certain extent. It is very promising in high-performance lithium battery electrode materials. Sun et al. [27] obtained Fe/Mn-MOF-74 by a one-step microwave method and further used it to synthesis hollow Fe-Mn-O/C microspheres. Endowed with the MOF-derived carbon-coated nanoparticle-assembled hollow structure, this metal oxide/carbon composite material can maintain 1294 mAh/g after 200 cycles at 100mA/g and behaved a reversible capacity of 521mAh/g at 1A/g, which is considered to have good cyclic stability and rate performance.

Generally, metal oxide/carbon composite materials can also be obtained by compounding flexible, strong, and conductive carbon materials with MOFs derivatives, such as carbon nanotubes, graphene, etc. Common strategies are surface coating or structuring three-dimensional conductive skeletons. Zhang et al. [28] used flexible carbon cloth with the characteristics of high mechanical strength and strong conductivity, and then grown MOF-derived ZnO@ZnO quantum dots/C core-shell nanorod arrays (NRAs) on it. This material obtained not only satisfies fast ion transfer but also has excellent mechanical strength and good corrosion resistance, so it has excellent rate capability and cycle stability in the laboratory test. Refer to data, it showed a reversible capacity of 1055 mAh/g at a current of 100 mA/g, and only 11% capacity got lost after 100 cycles at a current of 500 mA/g. Similarly, Yin et al. [29] employed a temperate coprecipitation method with ZIF-67 rhombic dodecahedron as a template and GO as a substrate, and successfully fabricated GO-MOFs derived rGO coating/sandwiching Co3O4. Due to the presence of the rGO coating, the electron transfer speed was improved and the volume expansion was alleviated, resulting in the material having excellent lithium storage performance (reversible capacity of 974 mAh/g at the current of 0.1 mA/g after cycling 100 times) and great cycle stability (the retention rate exceeded 95% after 100 cycles) (Fig. 4).

![Figure 4. The cycling performance and Coulombic efficiency of rGO@Co3O4.](image-url)
These all indicate that the compound of MOFs derivatives and carbon-based materials can play a synergistic role, which is an important development direction for the construction of new electrodes in the future.

4. Conclusion

MOFs material and its derivatives are considered as two promising families in the field of energy storage. Their high porosity, controllable chemical compositions and structures offer immense possibilities in searching for suitable anode material for LIBs. Although pristine MOFs material has surpassed the commercial graphite anode in terms of specific capacity, till now, its practical application is hindered by its high cost and low conductivity. Therefore, exploring topology structure with fast charge transfer is considered as the most urgent goal. Comparatively, MOF-derived materials, including porous carbon, metal oxide, and their composites, show much promise as anode active materials due to better rate capability and improved stability. Moreover, a series of strategies have been developed to strengthen their practical performances for meeting the requirements of next-generation batteries. At present, for many of them, their electrochemical performances still have great development prospect.

Overall, the utility of MOFs material or its derivatives as anode materials in LIBs is an emerging research topic, facing great challenges and opportunities. Throughout the future development of the MOFs as anode active materials, researches generally move in the direction of great rate capability and lower cost on the basis of high energy density. In terms of practice application, researchers favor to use methods such as modification, composite materials to address the stability and conductivity of LIBs to achieve the goal of commercialization.

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