

Review and prospect on Metal-organic framework-based Separators for Lithium–Sulfur Battery

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Abstract. With the continuous advancement of technology, wearable smart devices and electric vehicles (EVs) have become an integral part of our daily lives. The increasing demand for energy from these devices and vehicles has driven the pursuit of high-performance rechargeable battery technology. In particular, the widespread adoption of electric vehicles has set higher standards for the energy density and cycle life of batteries. However, the energy density of the widely used lithium-ion batteries is currently limited by the theoretical capacity of commercial cathode materials, and there are certain challenges in terms of safety and cost-effectiveness. Therefore, the development of a new generation of rechargeable batteries with higher energy density, better safety, and more economical cost is particularly urgent. Lithium-sulfur batteries, due to their high energy density, high economic benefits, and high environmental friendliness, are considered to be one of the most promising successors to the widely used lithium-ion batteries. However, in practical applications, the electrochemical performance of lithium-sulfur batteries still needs to be improved. In particular, the poor conductivity of sulfur/lithium and the shuttle effect of polysulfides make the cycle life of the battery less than ideal, and lithium-sulfur batteries (LSB) are still far from achieving large-scale commercialization. The separator used in lithium-sulfur batteries plays a crucial role in its cycle performance and safety. The current commercial separator lacks the ability to effectively regulate the shuttle of polysulfides and is prone to thermal runaway at high temperatures. Therefore, to improve these issues, various functional materials have been used to modify the separator. Among them, as a new type of porous coordination polymer, metal organic frameworks (MOFs) have become a promising material for modifying separators due to their large specific surface area and highly ordered tunable nano-holes. At the same time, their rich inorganic nodes and designable organic ligands allow for the customization of pore chemistry at the molecular level, which can interact with the electroactive components in lithium-sulfur batteries in a tunable manner. This article reviews the reaction mechanism of lithium-sulfur batteries and the structural advantages of MOFs in terms of pore chemistry and morphology and briefly introduces the research progress of MOF-based interfacial layers for the functionalization of LSB separators in recent years. It elaborates on the mechanisms by which various modified separators improve electrochemical performance and provides a prospect for the development prospects of the field.

Keywords: Lithium-sulfur batteries; MOFs; Separator.

1. Introduction

As the technology of intelligent unmanned driving advances, it is foreseeable that new energy vehicles, especially electric vehicles (EVs), will occupy a more important position in the field of transportation shortly. Therefore, electrochemical mobile energy storage technology with higher safety, high energy density, and cost-effectiveness is particularly important for the promotion of smart automobiles. Although, Lithium-ion batteries (LIBs) were proposed by Sony as early as 1991 and have been commercialized for use in our daily lives [1], the exploration for more portable, safe, and eco-friendly batteries has never ceased. This is mainly due to the unsatisfactory energy density of LIBs, the higher costs due to the abundance of lithium elements, and the potential safety risks caused by their electrochemical stability. The development of portable electronic devices and electric vehicles is forcing people to explore and develop rechargeable batteries with higher safety, high energy density, and cost-effectiveness. Lithium-sulfur batteries, as a promising energy storage system, have significant advantages. However, challenges in cycle stability and energy efficiency, especially

the penetration of lithium polysulfides, urgently require the improvement of separator materials. Metal-organic frameworks (MOFs) have become a research hotspot for Li-S battery separators due to their high specific surface area, adjustable pore structure, and good chemical stability.

The overall significance of this study lies in its potential progress in the field of energy storage, especially by developing new types of lithium-sulfur batteries with higher safety, high energy density, long cycle life, and cost-effectiveness, which can help alleviate the growing demand for electrochemical mobile energy storage.

1.1. Basic principles of lithium-sulfur batteries

Among the candidates for next-generation secondary batteries, lithium-sulfur batteries (LSBs) with sulfur cathodes and lithium anodes are considered promising due to their high theoretical capacity (1675 mAh/g) and energy density (2600 Wh/kg) [2]. Moreover, sulfur as the active material, has a high element content and can reduce the cost of large-scale production of lithium-sulfur batteries significantly, while its lower environmental impact and better overcharge protection performance [3] also make lithium-sulfur batteries highly commercially viable [4].

The working principle of lithium-sulfur batteries is based on the electrochemical reaction between lithium ions and sulfur. During discharge, lithium ions migrate from the anode (lithium metal) to the cathode (sulfur), reacting with sulfur to form lithium sulfide (Li_2S). The charging process is the reverse of this reaction. Unlike the lithium insertion/extraction mechanism in traditional lithium-ion batteries, LSBs utilize the limited element sulfur (S_8) and undergo a conversion mechanism.

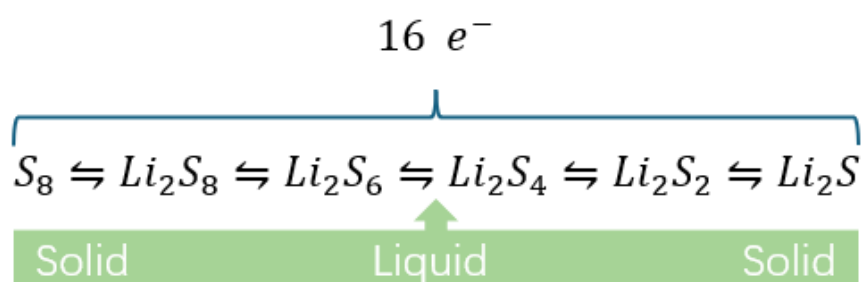


Fig 1. Charge discharge/electric reaction principle of lithium-sulfur batteries.

Generally speaking, the redox reaction in lithium-sulfur batteries involves a multiphase, multi-electron, and multi-step process [5]. During discharge, solid sulfur is sequentially reduced to various soluble intermediate products of lithium polysulfides (LiPSs), which are ultimately reduced to solid Li_2S by gaining electrons and breaking S-S bonds. As shown in Figure 1, the discharge reaction of lithium-sulfur batteries first forms soluble long-chain lithium polysulfides (Li_2S_n , $4 \leq n \leq 8$), providing 1/4 of the theoretical capacity (418 mAh/g). Then, it converts Li_2S_4 into short-chain $\text{Li}_2\text{S}_2/\text{Li}_2\text{S}$, providing 3/4 of the theoretical capacity (1255 mAh/g). It is worth noting that the produced long-chain lithium polysulfides can dissolve in the classical ether-based electrolyte, some of which may be reduced to $\text{Li}_2\text{S}_2/\text{Li}_2\text{S}$ on the Li metal anode surface through osmotic effect under the influence of a concentration gradient [6].

Unfortunately, due to poor electron/ion conductivity and high activation energy, these depositions are difficult to fully convert back into long-chain lithium polysulfides. This undesirable shuttle effect leads to rapid capacity loss and low Coulombic efficiency. Generally, the evolution of lithium polysulfides involves complex multiphase changes of solid-liquid-liquid-solid-solid. Although the solubility of lithium polysulfides is beneficial for solid-liquid conversion, the reaction oxidation kinetics in the final liquid-solid conversion are very slow, especially the final solid-solid conversion [7]. In other words, the formation rate of long-chain lithium polysulfides is faster than their consumption rate, leading to their accumulation in LSBs, especially severe in the case of dilute electrolytes and high sulfur loading. These unconverted lithium polysulfides can cause thickening of the electrolyte and further exacerbate the shuttle effect, leading to irreversible loss of active material and increased battery internal resistance, thereby deteriorating the overall performance of the battery.

1.2. The role of diaphragm

To curb the shuttle effect in lithium-sulfur batteries and enhance their electrochemical performance, researchers have proposed numerous solutions, such as cathode modification, electrolyte optimization, and separator modification.

Among these, the battery separator is distant from the electrochemical reaction interface but is in close contact with the electrodes. More importantly, it serves as a diffusion channel for lithium polysulfides (LiPSs). It not only isolates the anode and cathode to prevent direct electronic contact but also allows lithium ions to migrate between the electrodes. Thus, its structure and properties significantly influence the electrode reactions in LSBs and the shuttling of LiPSs between the two electrodes. It can be said that the separator's performance directly affects the battery's ionic transport efficiency, cycle stability, and safety. Therefore, an ideal separator should possess good chemical stability, sufficient porosity, and mechanical strength.

Generally, traditional separators, such as the commercially available polyolefin separators commonly used today [8], are typically composed of polymer porous membranes with large pores that do not interact with LiPSs. Hence, they struggle to suppress the shuttling of LiPSs between the two electrodes of LSBs. It is this unrestricted soluble lithium polysulfide migration through the separator that leads to an irreversible continuous loss of overall battery performance during charge-discharge cycles [9]. Therefore, while ensuring the basic functions of the polyolefin-based separator, if it is modified to reduce the separator pore size and give it the ability to adsorb and/or convert LiPSs, the "shuttle effect" of LiPSs would be suppressed effectively, which can significantly improve the overall performance of the battery [10].

2. Metal-organic Frameworks (MOFs)

Since the concept of Metal-Organic Frameworks (MOFs) was first defined by Yaghi's team in the 1990s [11], it has undergone rapid development and gradually become a hot research direction in the field of chemistry and materials. Over the past three decades, more than 20,000 MOFs have been constructed [12]. MOFs materials are characterized by diverse metal centers, controllable functional properties, high specific surface area, large porosity, and regular and adjustable pore size and shape structures, making them show huge application potential in the field of separators of LSBs. MOFs materials can be used as separators or separator coatings to improve ionic transport efficiency and suppress the shuttle effect of polysulfides, thereby significantly enhancing battery performance.

2.1. Overview of MOFs Materials

MOFs, also known as porous coordination polymers (PCPs), are two-dimensional or three-dimensional porous crystalline materials composed of metal cation salts or clusters and multi-toothed organic ligands. The structure of MOFs materials is flexible and can be assembled into different framework systems by changing the types of metal cations and organic ligands. Through the orderly arrangement of linkers and metal ions, MOFs can exhibit different structures. Because the types of organic ligands and metal ions are various, the structures produced by the orderly arrangement are theoretically almost infinite. Common organic ligands include carboxylate salts, phosphonates, sulfates, amines, phenols, etc., while metal ions mainly include transition elements and rare earth metals. Typically, the porosity of MOFs structures is greater than 50% of the MOFs crystal volume, and the surface area is usually between 1000 and 10,000 m²/g, exceeding the surface area values of traditional zeolites, activated carbon, mesoporous silica, and other materials. [13]

2.2. Potential of MOFs Materials

In theory, metal sites and polar functional groups on organic ligands have the potential to effectively adsorb polysulfides and prevent their migration within the cathode. [14] By fine-tuning the synthesis process, researchers can create precise and adjustable topological structures with specific functional unsaturated metal sites. In addition, the ordered porous skeleton of MOFs is

conductive to the rapid transport of Li^+ ions, promoting uniform electroplating and stripping of Li^+ ions on the anode side. Therefore, MOFs have the potential to serve as selective membranes for lithium batteries. By using MOFs as precursors to manufacture derivatives, the beneficial characteristics of MOFs-based materials can be retained while alleviating the issue of poor stability of MOFs materials.

3. Research progress on the application of MOF in lithium-sulfur battery separator

Recently, Metal-Organic Frameworks (MOFs) have become a focus of attention for researchers due to their unique structural features, such as highly ordered pore arrangement, adjustable pore size, and high porosity. [15] In particular, these characteristics of MOFs have shown potential application value in suppressing the polysulfide shuttling effect in lithium-sulfur batteries. [16] Uniform pore size distribution and large specific surface area provide MOFs with the ability to act as a physical barrier, effectively preventing the unwanted shuttling effect. Embedded metal elements in MOFs, such as nickel (Ni) and cobalt (Co), have significant catalytic properties and can react with soluble polysulfides as Lewis acids. [17] In addition, specific functional groups present in MOFs, such as sulfonic groups ($-\text{SO}_3\text{H}$) and amine groups ($-\text{NH}_2$), play a key role in restricting the migration of polysulfides through electrostatic interactions. [18] Therefore, integrating MOFs into the separator of lithium-sulfur batteries can significantly enhance battery performance.

3.1. Synthesis of MOF-modified diaphragm

The practicality of MOFs-based coating membranes mainly depends on their compatibility with the membrane substrate, which has a significant impact on the carrying capacity, mechanical properties, interface resistance, and thermal stability of the coating. Therefore, it is crucial to understand how to effectively combine the membrane with the MOF coating. Currently, there are mainly two methods of combination: one is the in-situ synthesis of MOFs directly on the polymer film; the other is a two-step composite pre-synthesis mixing method, in which MOF materials are first independently synthesized, then mixed with the polymer, and finally prepared into a film by casting or electrospinning technology [19].

3.1.1. In-situ growth method

Typically, in-situ synthesis of MOFs on polymer films can ensure that MOF particles are uniformly dispersed and firmly attached to the film, thus enhancing the compatibility between MOFs and polymers. This method can maintain the physical and chemical properties of MOF crystals while maximizing their surface activity.

Common synthesis methods include solvothermal synthesis and precursor gas phase transformation technology. Solvothermal synthesis requires adjusting the reaction time and temperature according to the specific MOF material to ensure the structural stability of the MOF and the separator. The gas phase transformation technology involves dispersing metal ions in the matrix material and then allowing the organic ligands to diffuse into the precursor under gas phase conditions to trigger the self-assembly process. For example, Manthiram et al. [20] reported an in-situ synthesis method of Co-MOF on a Celgard separator, after sulfiding treatment, the Co_9S_8 formed a tight physical bond with the separator, demonstrating excellent mechanical stability and flexibility. The polar hollow array of Co_9S_8 not only has a regular arrangement but also is uniformly distributed, providing a larger contact area, while reducing the loading of the coating, effectively reducing the internal resistance of the battery. Internal resistance of the battery.

3.1.2. Pre-synthesized MOF mixing method

Fabricating MOF-modified separators can be achieved by integrating pre-synthesized MOF particles with a matrix material. This approach helps to balance the sieving properties and permeability of MOF particles. To address the brittleness issues of MOF crystalline thin films and

the challenge of inter-particle gaps that cannot effectively block polysulfides, filling the gaps between MOF particles with polymers and inorganic fillers has been proposed [21]. The fillers in the composite separator, together with MOFs, play a crucial role in preventing the shuttling of polysulfides. Moreover, ensuring the compatibility between the fillers and MOFs is essential for the formation of an effective barrier layer. Current research on the hybrid production of MOFs-modified separators mainly involves two methods: mechanical stirring and vacuum-assisted filtration.

The mechanical stirring method involves mixing pre-synthesized MOF particles with polymers and other fillers, dissolving them in an appropriate organic solvent, and then forming a thin film through a doctor-blade process. On the other hand, vacuum-assisted filtration uses MOF particles as a filtration medium, adding binders or carbon materials on the surface of MOF particles to form a three-dimensional structured membrane. The physical bonding between the MOF substrate and filtration materials facilitates interfacial compatibility. This method not only avoids potential damage to the MOF structure during mechanical stirring and mixing but also allows for precise control of the film thickness, simplifying the film fabrication process, which is more conducive to the application of lithium-sulfur batteries.

Zhou et al. [22] proposed a vacuum-assisted filtration process, using HKUST-1 as the building unit and PVDF as a binder, to fabricate a MOFs-modified separator. The in-situ formation of the MOF film on a Celgard 2400 separator was achieved through a hydrothermal synthesis method, which involves pumping the precursor solution onto the separator for self-assembly. Subsequently, PVDF was dissolved in a solvent and uniformly coated onto the MOF particles through pumping, enhancing the adhesion between particles and filling the gaps between them. By repeating this process, the gaps between MOF particles were densified, resulting in a separator with higher flexibility. Experimental results show that MOF particles are evenly distributed on the surface of the separator, and the pores mainly originate from the structural characteristics of the MOFs themselves. Compared with MOFs directly grown on the PP separator, this method provides better thickness control. Although some micropores of MOFs may be blocked by PVDF during the filtration process, leading to a reduction in surface area and the number of pores, the pore size distribution still maintains relative consistency and a narrower range compared to the original MOF film.

3.2. Performance Improvement

3.2.1. Inhibition of shuttle effect

The sluggish kinetics of the conversion from sulfur (S) to lithium sulfide (Li_2S) can exacerbate the shuttle effect and polarization losses. Therefore, to curb the shuttle effect, it is imperative to regulate the reactivity of sulfur. Catalysis has been proven to be an effective approach [23]. Many catalytic materials, such as heteroatom-doped carbons, metal compounds, and single-atom catalysts, have been reported [24]. Due to their hybrid valence metal centers or chemically active organic linkers, MOFs can serve as catalytic materials. For instance, studies have shown that introducing nickel into the metal-organic framework (MOF) material ZIF-8 can significantly enhance the catalytic activity of polysulfides. It is indicated that the doped nickel can accelerate the redox reaction process of polysulfides. In MOFs, defects and vacancies often act as active centers for catalytic reactions, a characteristic that is relatively easy to achieve during the synthesis of MOFs [25].

The chemical properties of the porous structure are crucial for mass transfer and ionic migration performance. By accurately controlling the pore size of metal-organic frameworks (MOFs), selective filtration of ions of different sizes can be achieved, thereby regulating their transport efficiency. Considering the difference in ionic charge, the migration behavior of ions can be effectively modulated by altering the charge state within the pores of MOFs. Particularly, MOFs with cage-like pore structures have attracted significant attention due to their unique properties. This structure can encapsulate molecules or ions within its cavity, effectively restricting the migration of sulfur and polysulfides, which helps to suppress the shuttle effect in batteries.

3.2.2. Limiting the growth of lithium dendrites

Since the control of polysulfide migration within the battery, the deposition process of lithium ions on the anode often exhibits irregularity. During the deposition, lithium ions tend to grow rapidly along the tip areas, and this growth rate usually exceeds their radial expansion, ultimately leading to the formation of one-dimensional fibrous lithium dendrite structures [26]. This dendritic precipitation, coupled with significant volume changes, results in the formation of "dead lithium," which leads to increased polarization and capacity loss [27]. In addition, the abnormal growth of lithium dendrites has the potential to penetrate the battery's separator, posing a serious safety hazard. The excessive proliferation of lithium dendrites not only affects the battery's performance but may also cause internal short circuits due to their sharp shape, thereby increasing the risk of thermal runaway [28]. It is widely believed that the formation of lithium dendrites is due to the non-uniform spatial distribution of lithium ions, resulting in a lack of uniformity in the deposition process of lithium metal. Some studies suggest that using MOFs as a surface coating for the battery separator can enhance the uniformity of lithium flux. This approach helps to optimize the deposition pattern of lithium ions, thereby forming a smoother and more consistent lithium metal layer on the battery anode [29].

4. Summary and Outlook

Since the innovative material MOFs were first considered for potential application in lithium-sulfur battery technology, their customizable structures have demonstrated unique advantages in addressing the challenges of LIBs. Recently, significant progress has been made in lithium-sulfur battery technology based on MOFs. Nevertheless, to further enhance battery performance, an in-depth investigation of the interaction mechanisms between MOFs and electroactive materials is essential. This opens endless possibilities for future research in the field of LSBs.

Metal-organic framework materials have provided new ideas for the research of lithium-sulfur battery separators, showing great potential to improve battery performance with their distinctive characteristics. Future research should focus on the design optimization and engineering application of materials to achieve the widespread commercialization of LSBs.

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