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ARTICLE TYPE

# Enhanced Electrochemical Performance of Sulfur Cathodes with a Water-Soluble Binder

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To improve the electrochemical performance of lithium-sulfur batteries, LA132, a kind of polyacrylonitrile is used as a water-soluble binder for sulfur cathodes. The optimal content of LA132 binder is investigated by electrochemical tests and morphology characteristics. Galvanostatic charge-discharge tests show that sulfur cathodes with 5 wt% LA132 (relative to the mass of whole cathode materials) exhibit a significant improvement in discharge capacity and cycle performance compared with the ones using 10 wt% polyvinylidene fluoride (PVDF) and LA132. Cyclic voltammetry and electrochemical impedance spectroscopy confirm that the LA132 sulfur cathodes show lower resistance and better kinetic characteristics. The effect of LA132 binder is further investigated *via* visual picture and scanning electron microscopy (SEM). The visual picture shows the decreased bond strength of PVDF caused by its swelling, leading to a loss of active materials from the current collector. The morphologies of sulfur cathodes before and after 100 cycles indicate that the non-swellable LA132 could stabilize porous structures of cathodes during cycling and enhance the electrochemical performance of sulfur cathodes.

## Introduction

Lithium-sulfur (Li-S) batteries are considered to be one of the most attractive secondary battery systems. Assuming that the lithium and sulfur are completely reacted into $Li_2S$, the specific capacity and energy density of Li-S batteries are as high as 1675 mAh $g^{-1}$ and 2600 Wh $kg^{-1}$, respectively.[1-3] Either the sulfur electrode itself or the intermediate products formed during the cycling process are seldom harmful to the environment. Moreover, sulfur is abundant and low cost.[4] Thus, Li-S batteries can well meet the requirements of future power.[5] Nevertheless, there are still some drawbacks in sulfur cathodes that restrict the further development of Li-S batteries.[6-8] Firstly, the insulating nature of elemental sulfur ($5\times10^{-30}$ S $cm^{-1}$, 25 °C) leads to the low utilization of the active materials. Secondly, lithium polysulfides formed as the intermediate products of sulfur cathodes are readily dissolved into the organic electrolyte and diffuse to Li anode, generating "shuttle effect" and causing the loss of active materials.[3]

Many efforts have been taken to solve these problems. The main researches are focused on the design of conductive structure, optimization of electrolyte composition and the anode protection.[9-15] Recently, some studies are concentrated on the binders used in sulfur cathodes.[16-19] The fundamental effects of binders in lithium-ion batteries involve binding the electrode materials on the current collector and ensuring good electrical contact between the active materials and the conductive agents.[20] When it comes to Li-S batteries, a suitable binder not only needs a strong adhesion strength but also needs to have an ability to buffer the volumetric expansion during cycling and stabilize the cathode structure.[21] Furthermore, it is ideal for binders to restrain the dissolution of lithium polysulfides into electrolyte.[22]

Polyvinylidene fluoride (PVDF) is one of the most popular binders used in Li-S batteries due to its strong adhesion strength, high chemical and electrochemical stability.[23] However, some intrinsic natures of PVDF take a negative impact on the electrochemical performance of Li-S batteries.[24-25] For instance, PVDF is prone to swell and dissolve in the organic electrolyte at high temperature, leading to a collapse of conductive network and high interface resistance. On the other hand, N-methyl pyrrolidinone (NMP) with a high boiling point is used as a solvent to dissolve PVDF. Therefore, an elevated temperature of 80 °C is essential to dry the cathode, causing the loss of active materials. In addition, the environmental problems caused by the toxic NMP cannot be ignored. More recently, aqueous binders have been draw more and more attention as a promising binder system for Li-S batteries due to their low cost, environmentally friendly and low boiling point.[26] Water-soluble gelatin has been successfully used as a binder in the Li-S batteries due to its multifunctional effects on the sulfur cathode.[27] Wang[28] reported the styrene butadiene rubber-sodium and carboxyl methyl cellulose (SBR-CMC) with strong adhesion property and dispersion ability as an aqueous binder in Li-S batteries. Carbonyl-β-cyclodextrin (C-β-CD) was also used in sulfur cathodes as a binder. Its strong bonding capability, high solubility in water and electrochemical stability contribute to the enhanced cycle performance of Li-S batteries.[29]

LA132 ($\text{-[-}R_1\text{-}R_2\text{-}CH_2\text{-}CH(CN)]_n\text{-}$) is also a kind of aqueous binder with strong adhesion property due to the strong polar group -CN in the main chain segment of its structure. It has been successfully used in the preparation of many anodes and cathodes in Li-ion batteries, such as $LiFePO_4$ and Si.[30-31] Herein, we proposed LA132 to use in the fabrication of Li-S batteries with different additive content (5 wt% and 10 wt%). The active carbon and sulfur composite (AC-S) was prepared as active materials in cathodes. The porous active carbon with high specific surface facilitates electronic/ionic transport and is prone to absorb the polysulfides. So it is significantly important for binder to accommodate the volume change during charge-discharge process and stabilize the electrode structure. The effects of LA132 binder on the specific capacity and capacity retention of the AC-S composite cathodes were investigated, which were superior to PVDF binder and exhibited great potential for Li-S batteries.

## Experimental Section

### Preparation of electrode and cell assembly

The AC-S composite was prepared *via* heating the mixture of active carbon (AC) and sulfur powder (S) (1:1 by weight) in a seal container filled with $N_2$ at 155 °C for 10 h. Afterwards, the AC-S (active material), acetylene black (conductive agent) and PVDF (binder) with a weight ratio 7: 2: 1 were mixed with NMP to form an uniform slurry. Subsequently, the slurry was casted on the aluminum foil and dried at 80 °C for 12 h. When LA132 was used as the binder, the ratio was adjusted to 7: 2: 1 (10 wt%-LA) and 7: 2: 0.47 (5 wt%-LA), respectively. Distilled water was used as solvent and the drying temperature was 60 °C. The electrodes were denoted as 10 wt%-PVDF, 10 wt%-LA, and 5 wt%-LA, respectively. Each current collector contained ~2 mg $cm^{-2}$ active materials.

Coin-type (CR2016) cells were assembled in an Ar-filled glovebox with $H_2O$ and $O_2$ content lower than 1 ppm. Sulfur electrode was used as the cathode, microporous polypropylene (PP) membrane as the separator and lithium foil as the anode. The electrolyte was composed of 1, 2-dimethoxyethane (DME) and 1, 3-dioxolane (DOL) (V/V=1:1) containing 1 mol $L^{-1}$ bis-(trifluoromethane) sulfonimide lithium (LiTFSI), 0.1 mol $L^{-1}$ $LiNO_3$. The electrolyte amount in each cell was 80 µL $mg^{-1}$.

### Characterization

The structures of materials were characterized by X-ray diffraction measurement (XRD, Bruker-AXS D8 DISCOVER using Cu $K_\alpha$ radiation). Thermal gravimetric analysis (TGA) was conducted under a $N_2$ atmosphere at a heating rate of 10 °C $min^{-1}$ from 30 to 700 °C on a TG-DSC instrument (NETZSCH STA 409 PC). The morphologies of the sulfur cathodes before and after charge-discharge tests were observed with a scanning electron microscopy (SEM) (Hitachi S-4800). Before SEM measurement, the remaining soluble polysulfide in the cathode was completely washed with DME in an Ar-filled glove box.

Cyclic voltammetry (CV) measurement was performed between cutoff potentials of 1.0 to 3.0 V (*vs.* $Li/Li^+$) at a scan rate of 0.2 mV $s^{-1}$ with a CHI 660A electrochemical workstation (Chenhua, China). Electrochemical impedance spectroscopy (EIS) was measured with the cells at open circuit voltage. The frequency range applied was from 100 kHz to 10 mHz with a potentiostatic signal amplitude of 5 mV. The galvanostatic charge-discharge tests were carried out in the range of 1.5-3.0 V (*vs.* $Li/Li^+$) at a current density of 0.5 C (1 C= 1675 mA $g^{-1}$) based on the mass of sulfur with cell testing machine (LAND Electronic Co. CT2001A, China). All experiments were conducted at room temperature.

## Results and discussion

XRD patterns of the AC, S and as-prepared AC-S composite are presented in Fig. 1a. AC exhibits two broad diffraction peaks around 24° and 44°, corresponding to (002) and (101) diffractions of graphitic carbon.[32-33] The entire disappearance of the diffraction peaks from crystalline sulfur in XRD pattern of AC-S demonstrates that sulfur has been successfully impregnated into the AC matrix.

The sulfur content of the AC-S composite was ascertained by TGA under a $N_2$ atmosphere from 30 °C to 700 °C with a heating rate of 10 °C $min^{-1}$ (Fig. 1b). The weight of the AC-S composite decreases with increasing temperature from 300 °C and the weight loss is continuous until the AC-S composite are heated to over 500 °C, which indicates that the reduction in weight of the AC-S composite is due to the evaporation of sulfur. The sulfur content is 50.6% in the AC-S composite, which is consistent with the mixture weight ratio in the experimental section.

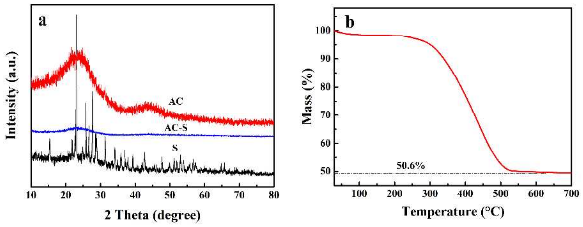

**Fig. 1** (a) XRD patterns of S, AC and AC-S composite, (b) TGA curve of AC-S composite.

CV curves of the sulfur cathodes with different binders at a scan rate of 0.2 mV $s^{-1}$ are shown in Fig. 2. All curves show two distinct reduction peaks and one oxidation peak, which are in accordance with the typical CV characteristics of the sulfur cathodes.[3] Furthermore, the reduction and oxidation peaks of the LA132 cathodes appear much sharper than those of the PVDF cathode and the voltage difference (ΔE) between oxidation and reduction peaks for LA132 cathodes are also smaller. Both of the sharper peaks and the lower ΔE demonstrate that the redox reactions in the LA132 cathodes behave more likely as a Nernst system.[28, 34]

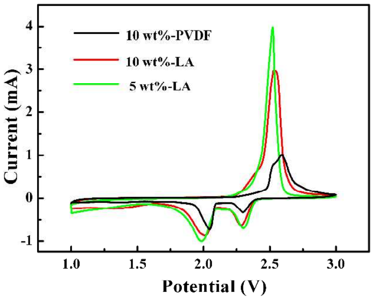

**Fig. 2** Typical CV curves of the sulfur cathodes with different binders at a scan rate of 0.2 mV $s^{-1}$

The cycling performances and coulombic efficiencies of cathodes with different binders are compared in Fig. 3a. It can be seen that the discharge capacities of three sulfur cathodes decrease gradually with the increasing cycle numbers. The addition of $LiNO_3$ increases the coulombic efficiencies of three cathodes to nearly 100%.[35]

In case of the LA132 binder, the discharge capacity of 5 wt%-LA and 10 wt%-LA at the first cycle at 0.5 C are 901.6 mAh $g^{-1}$ and 678 mAh $g^{-1}$, respectively (All of the capacities mentioned in this report are calculated based on the mass of sulfur). After 100 cycles, the reversible capacity still remains at 470 mAh $g^{-1}$ and 419.4 mAh $g^{-1}$. In contrast, the discharge capacity of PVDF drops from the initial 708 mAh $g^{-1}$ to 322.7 mAh $g^{-1}$ with a capacity retention rate of 45.6% after 100 cycles. Comparing with the three cathodes, we can conclude that both the LA132-based cathodes have better electrochemical performance than the PVDF-based cathodes in terms of the capacity retention and the discharge capacity. When it comes to the LA132-based cathodes, the content of LA132 in the cathode has a significant impact on the performance of Li-S batteries. As the LA132 is non-conduct, the 5 wt%-LA cathode has a lower internal resistance giving rise to a higher sulfur utilization. In addition, the electrochemical performance of cathode with 5 wt% PVDF has been exhibited in supplementary information (Fig. S1).

Fig. 3b shows the charge-discharge profiles of Li-S batteries using 10 wt%-LA, 5 wt%-LA and 10 wt%-PVDF cathodes after 100th cycles. Typical two-plateau behavior is observed during discharge, which could be assigned to the formation of long-chain polysulfides (around 2.3 V) and $Li_2S_2/Li_2S$ (around 2.0 V).[36-37] As seen, the 5 wt%-LA sulfur cathode shows a lowest charge plateau potential and highest discharge plateau potential than those of the 10 wt%-PVDF and 10 wt%-LA sulfur cathodes. Compared with PVDF, the smaller potential separation between the charge and discharge plateaus indicates better kinetic characteristics of the sulfur cathodes using LA132 as the binder.

Furthermore, LA132 binder has been applied in cathodes with high sulfur loading (49 wt%) and different carbon hosts (Fig. S2, 3). Better cycle performances have been achieved comparing with the cathodes with PVDF binder.

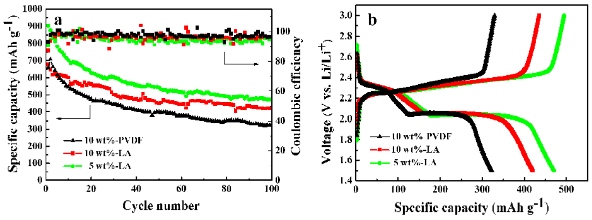

**Fig. 3** (a) Cycle performances and coulombic efficiencies of sulfur cathodes with different binders at 0.5 C, (b) Galvanostatic charge-discharge profiles of sulfur cathodes with different binders at 0.5 C after 100 cycles.

In order to gain the insight into the effect of LA132 binder on sulfur cathodes, Fig. 4 shows typical Nyquist plots of the sulfur cathodes with different binders before cycling and after 100 cycles. It can be seen from Fig. 4a that the Nyquist plots before cycling are composed of a semicircle at high frequency and medium frequency and an inclined line in the low frequency region. The high-frequency intercept on the real axis represents the ohmic resistance of the cell, including the electrolyte and electrode resistances. The semicircle at high to medium frequency is attributable to the surface layer and interfacial impedance of the electrodes, and the line is due to the Li-ion diffusion within the cathodes.[38-39] Before cycling, both the 10 wt%-LA and the 5 wt%-LA sulfur cathodes show a lower resistance than the PVDF cathode, indicating a low internal resistance of LA132 binder, resulting in better cell performance. After 100 cycles, there are two semicircles as shown in Fig. 4b. The semicircle in the high frequency region should reflect the charge transfer process at the conductive agent interface. The semicircle in the middle frequency range could be attributed to the formation of $Li_2S/Li_2S_2$ on the carbon matrix in the cathodes.[40-41] The equivalent circuits are shown in the insets of Fig. 4a, b. It is obviously seen that both the charge-transfer resistance and the interface resistance of the LA132 electrodes are much lower than those of the one with PVDF. This can be inferred that the swelling of PVDF impair the electrical contact between the cathode materials and current collector, causing the increase of the contact resistance and decreasing the charge-transfer ability. Meanwhile, the swelling of PVDF leads to the collapse of the porous conductive network giving rise to the agglomeration of irreversible $Li_2S_2/Li_2S$ on the cathodes, resulting in the higher interfacial resistance. While the non-swellable LA132 is used as binder, it plays a buffer role during the volume change of the cathodes and maintains a maximum interfacial area for charge transfer.

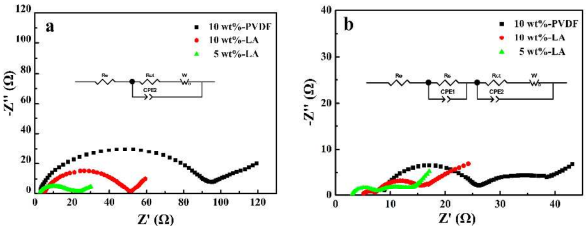

**Fig. 4** Nyquist plots for the sulfur cathodes in the frequency range of 100 kHz-10 mHz: (a) before cycle, (b) after 100 cycles. Inset: the equivalent circuits.

To further explore the effect of LA132 binder, the macrocosmic surfaces of different cathodes were first investigated after 100 cycles. The visual picture of separators and sulfur cathodes is shown in Fig. 5. As seen in the Fig. 5, the brown colour on separators is related with the $Li_2S_x$ diffusing from the cathodes.[42] Quite a lot of active materials fall off from the current collector and a few cathode components are stuck to the separator, whereas the cathodes with LA132 binder don't stick any components to the separators and the surfaces of cathodes are undamaged (Fig. 5b, c). This phenomenon demonstrates the viewpoint we have discussed above: PVDF could swell or even dissolve into liquid electrolyte, and it easily forms a gel during cycling, which decreases its adhesion ability and then causes a loss of active material. In contrast, the strong adhesion property and insolubility of LA132 in the liquid electrolyte contribute to the integrated surfaces of sulfur cathodes.

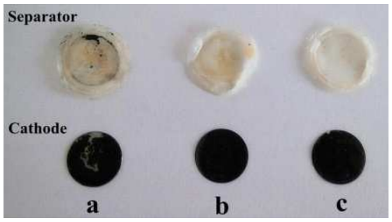

**Fig. 5** Visual picture of separators and sulfur cathodes after 100 cycles: (a) 10 wt%-PVDF cathode, (b) 10 wt%-LA cathode, (c) 5 wt%-LA cathode.

The microcosmic surfaces of cathodes were observed by SEM before cycling and after 100 cycles to demonstrate the relationship between morphologies and electrochemical performance of sulfur cathodes with different binders (Fig. 6). As we can see, all the cathodes hold a porous-like structure in a fresh state. However, the distribution of cathodes with the two different contents of LA132 is more homogeneous than that with PVDF. The well dispersion of acetylene black and active materials can enhance the electrical conductivity of the cathodes. The surfaces of different cathodes after 100 cycles were also showed in the Fig. 6 (a2, b2, c2). All the surfaces of the cathodes became denser and the growth of particles in cathodes is obviously observed. The denser film is the final product $Li_2S_2/Li_2S$, according to previous reports.[28, 43] However, the surface of PVDF cathode is much denser than that of LA132 cathodes, indicating the electrochemical reaction in PVDF cathodes is more irreversible. When PVDF is used as binder, it readily forms a gel in the cathode and leads to the poor contact with current collector and the collapse of the conductive network, both of which decrease the electrical conductivity of cathodes and accelerate the agglomeration of irreversible $Li_2S_2/Li_2S$.[44]

When it comes to LA132 cathodes, the 5 wt%-LA cathode holds the smaller particles after 100 cycles than 10 wt%-LA cathode. As we know, the addition of binder in electrodes will increase the internal resistence of electrodes and reduce the contact surface between electrodes and electrolyte.[45] Too much amount of binder may contribute to the agglomeration of irreversible $Li_2S_2/Li_2S$ phase with increasing cycles. We believe that 5 wt%-LA cathodes with the least amount of non-conducting component are beneficial to the electrochemical reaction and then the formation of irreversible $Li_2S_2/Li_2S$ film can be alleviated. This phenomenon is also in accordance with the electrochemical performance of sulfur cathodes with different binders.

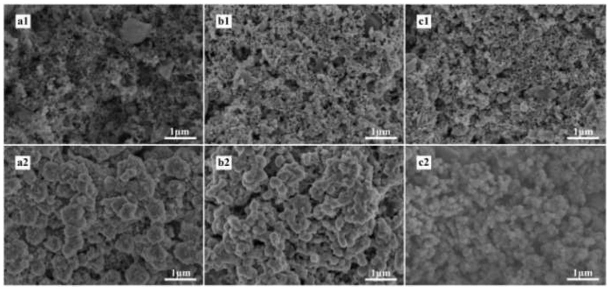

**Fig. 6** SEM images of the sulfur cathodes: the 10 wt%-PVDF cathode before cycle (a1) and at the end of the 100th charge (a2), the 10 wt%-LA cathode before cycle (b1) and at the end of the 100th charge (b2), the 5 wt%-LA cathode before cycle (c1) and at the end of the 100th charge (c2).

The morphology of AC-S and the distribution of sulfur in AC before and after 20 cycles are confirmed by TEM and STEM-EDX (Fig. S4, 5). Clearly, carbon and sulfur elements are detected across a selected area of the AC-S samples, confirming the existence of sulfur in the AC-S composite. In particular, the sulfur shows homogeneous distribution before and after 20 cycles.

The characteristics of cathodes with two binders in Li-S batteries are elucidated by the schematic diagram (Fig. 7). The high specific capacity and superior cycle performance of LA132 sulfur cathodes are closely related to the different properties of LA132 compared with PVDF in the electrolyte. PVDF is gelled by the organic electrolyte during cycling. The absorption of solvent may weak the bonding role of the binder, giving rise to the loss of cathode materials. Moreover, conductive network are readily collapsed under the combined impact of the swollen PVDF and the expanded active materials. On contrary, when the non-swellable LA132 is used as binder, it exhibits larger adhesive strength and smaller solvent absorption than PVDF, stabilizing the electrode structure and enhancing the electrochemical reaction of Li-S batteries.

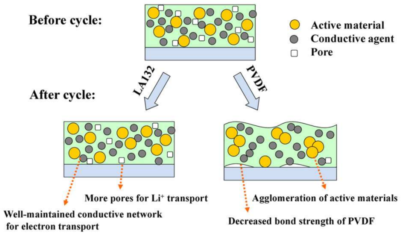

**Fig. 7** Schematic structure for the sulfur cathodes before and after cycling with different binders.

## Conclusions

We have successfully applied water-soluble LA132 as a binder for Li-S batteries. The 5 wt%-LA132 cathode exhibits a reversible capacity of 470 mAh $g^{-1}$ at 0.5 C after 100 cycles, showing a remarkably improved cyclability as compared with the traditional PVDF cathode. Electrochemical tests indicate that the LA132 sulfur cathodes have smaller internal resistance and better kinetic characteristics than those of PVDF cathodes. SEM images further confirm that LA132 binder could suppress the agglomeration of $Li_2S_2/Li_2S$ and maintain the porous structure of the sulfur cathode. We can conclude that non-swellable LA132 binder not only functions as a high adhesion agent and a strong dispersion agent, but also plays an important role in stabilizing the structure of cathodes. What's more, LA132 binder with water as solvent has advantages of environmental friendliness, low cost and safety. Based on this work, we can have an outlook that water-soluble binder such as LA132 is a promising kind of binders in Li-S battery applications.

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## Notes and references

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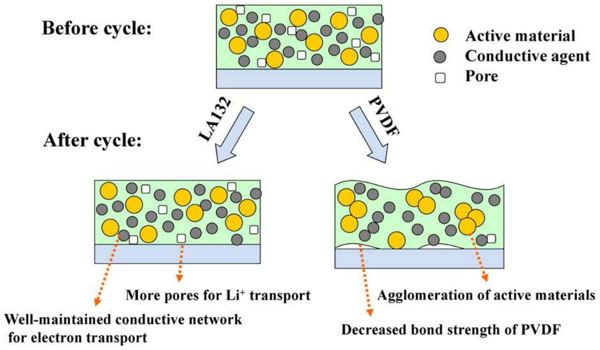
# TOC



The LA132 is a promising binder for Li-S batteries due to its strong adhesion ability and non-swellable property.