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Electrostatic Shield Effect: An Effective Way to Modify Celgard Membrane Separator Used in Lithium-sulfur Battery

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As one of the most potential power sources and energy storage systems, the lithium–sulfur battery (LSB) has received intense interest because of its considerable theoretical energy density, lower cost and eco-friendliness. But the commercial application of LSB is still impeded by the fast capacity decay resulted from the polysulfide shuttle, which is one of the primary factors responsible for the degradation of capacity. Herein, we demonstrate a strategy to trap polysulfide within the space between cathode and separator: a composite separator consists of lithiated polystyrolsulfon acid (Li-PSSA) and alginic acid (AA) are used to modify the surfaces of both cathode and separator based on electrostatic shield effect. By this specially design, the negatively charged SO_3^- groups on Li-PSSA function as an electrostatic barrier for soluble polysulfide through coulombic repulsion, whereas the Li^+ groups on Li-PSSA can provide the necessary paths for lithium ions to transmit within LSB. The selective shielding effect shows an effective suppression for the shuttle effect of polysulfide by confining them within the cathode side. This strategy has greatly improved the cyclic stability of LSB, presenting an ultralow capacity decay of 0.141% per cycle after 100 cycles at 0.1 C, far superior to that of the battery without any modification. Therefore, this simple strategy could be an alternative approach for LSB application.

Keywords: Lithium-sulfur batteries; Electrostatic shield effect; Separator; Shuttle effect; Cyclic stability.

1. INTRODUCTION

With the high theoretical specific capacity of 1675 mAh/g and energy density of 2600 Wh/kg, the lithium–sulfur battery (LSB) has become one of the most expected candidates for Lithium-ion battery (LIB), attracting large attention among the researches [1, 2]. In addition, the S element is abundant, low cost and has environmental friendliness, which makes LSB more suitable for practical application and commercialization [3]. However, there are still a number of drawbacks not solved before LSB’s commercial application [4]. Firstly, the specific discharge capacity of LSB is mainly decided by the utilization of active materials in the cathode, but the insulation of S and its discharge products (Li_2S) limits the LSB’s virtual capacity. Secondly, the diffusion and shuttle of polysulfide have caused the LSB’s low efficiency and short cyclic life. The highly soluble long-chain polysulfide (Li_2S_X, $4 \le x < 8$) can migrate through the separator easily and lead to the loss of active materials, which is severely responsible for the capacity fade [5, 6].

To reduce the so-called shuttle effect, researches have focused on the modification of the cathode [7]. To trap the polysulfide within the cathode region, many researchers tried to restrict sulfur in the pores of carbon materials or the conductive polymer structure [8, 9]. Different carbon materials including microporous carbons, carbon nanotubes/fibers, and carbon spheres; different polymers such as polypyrrole and poly(3,4-ethylenedioxythiophene), have been regarded as sulfur-host cathodes and had a positive effect on reducing the shuttle effect of polysulfide and increasing the cyclic performance of LSB [10,11]. Another approach to block the shuttle effect is to introduce various coating to the surface of cathode or separator, which can immediately cut off the diffusion of polysulfide to the anode [12,13]. But in most cases, single modification of cathode or separator can only block the polysulfide at a specific space weakly, especially the modification on the surface will cause the wrinkle of separator.

In this work, herein, we report a composite separator based on commercial Celgard membrane modified using lithiated polystyrolsulfon acid (Li-PSSA) and alginic acid (AA). The negatively charged SO_3^- groups on Li-PSSA can block soluble polysulfide through coulombic repulsion. Moreover, the coating of Li-PSSA@AA changed the polarity of separator’s surface from hydrophobic to hydrophilic, which improved its electrochemical performance inside the cell. By using the as-made separator, the specially designed LSB’s structure can not only effectively reduced the shuttling of polysulfide, but also transform the surface of the separator from hydrophobic to hydrophilic. Consequently, this strategy has greatly improved the cyclic stability and capacity retention of LSB, demonstrating a respectively superior performance compared with pristine battery without any modification inside.

2. EXPERIMENTAL

2.1. Material preparation

After the lithiation of PSSA (ALDRICH 434574; average Mw~1000000, powder) and AA (Aladdin S100126), they were mixed in deionized water to get homogeneous and different content of 2 wt%, 5 wt%, 8 wt% (ratio of AA to PS: 5 wt%) Li-PSSA@AA solution.

A Polypropylene（PP） microporous monolayer separator (Celgard 2500) with a thickness of 25μm and a pore size of 0.064 μm was used as a pristine cell separator and a support for the modification. First the pristine separator was pretreated for 45 min in the mixed solution of $K_2Cr_2O_7$ and H_2SO_4 at 60 °C to improve the hydrophility of Celgard surface. The ratio of $K_2Cr_2O_7$ to H_2SO_4 was 4 wt% and the content of H_2SO_4 was 8 mol/L [14]. The composite separator was fabricated by carefully coating the mixed solution of Li-PSSA@AA on one side of the pristine separator through scrape coating, and then it was transferred into an air oven, dried at 50°C for 12 h. The final composite separator was uniformly cut into circular disks, transferred into the glove box, and used for LSB's assembly.

The slurry of cathode material was prepared by ball milling 70 wt% of sublime sulfur (Aladdin S106611; AR 99.5%), 20 wt% of carbon black (Super P, Timcal) and 10 wt% of polyvinylidene fluoride (PVDF) binder in *N*-methyl-2-pyrolidone (NMP) for 24 h. Then the slurry was casted onto Al foil-current collectors and dried for 12 h at 50 °C in a vacuum oven to get the pure sulfur cathode. The modified cathode was fabricated by coating the mixed solution of Li-PSSA@AA on the surface of pure sulfur cathode with a scraper and then dried for 12h at 50°C in a vacuum oven according to literature [15]. Both pure sulfur cathode and modified cathode were cut into circular disks with a diameter of 14 mm and transferred into the glove box for the assembly of LSB. The sulfur loading in a fresh cathode disk of this work is 1.5–1.8 mg/cm^2.

R2025-type coin cells were assembled in an argon-filled glove box with lithium metal as the anode, using 1M lithium *bis*-trifluoromethanesulfonylimide (LiTFSI) in *1,3*-dioxolane (DOL)/dimethoxyethane (DME) (1/1, v/v), including 0.1 M $LiNO_3$ as the electrolyte. 50μL of electrolyte was added per coin cell. The assembled LSB cells were set for 12h at 25 °C before electrochemical cycling.

2.2. Characterization methods

The morphology of the samples was conducted by scanning electron microscopy (SEM, JSM-6380). The distribution of elements and radicals on their surface were characterized by energy dispersive X-ray spectrometers (EDS, ISIS-300) and FTIR spectrophotometer (IR-prestige21, Shimadzu Co., Japan), while the spectra were acquired at 4 cm^{-1} resolution in the 600 to 4000 cm^{-1} region. The hydrophility of different separators were characterized by the contact angle instrument.

2.3. Electrochemical measurements

Galvanostatic charge–discharge tests were measured within the potential range of 1.5-2.8 V versus Li/Li$^+$ at different current densities. The tests were finished using a CT2001A cell test instrument (LAND Electronic Co. Ltd., China). All specific capacity values were calculated on the basis of sulfur content. Electrochemical impedance spectroscopy (EIS) was performed via the Solartron 1255 B frequency response analyzer coupled with a Solartron 1287 electrochemical interface in the frequency range from 100 MHz to 0.1Hz with an applied voltage of 5 mV. All the electrochemical tests were carried out at room temperature [16].

3. RESULTS AND DISCUSSION

3.1 Structure and morphology observation

Firstly, The thickness and weight of different separators were measured and the data was listed in Table 1. Celgard 2500 (No 1 , PP) was used without any modification. The sample No 2 was

Celgard 2500 treated with $K_2Cr_2O_7/H_2SO_4$ solution. And the sample No 4 was based on Celgard separator and was coated with Li-PSSA@AA solution. It is shown in Table 1 that after the pretreatment of $K_2Cr_2O_7/H_2SO_4$, the weight of separator didn't change obviously. After coated with the solution of Li-PSSA@AA, the weight of composite separator increased with increasing the concentration of Li-PSSA@AA instead. The thickness of coated layer can be readily adjusted by controlling the concentration of Li-PSSA@AA solution.

From Figure 1 we can see the FTIR spectra of the composite membrane coated with Li-PSSA@AA. As shown in this figure 1, the peaks of 2920 and 1370 on the first curve correspond to the radical groups on the surface of polypropylene (PP), and the peaks of 3416 and 1178 on other curves correspond to the radical groups on the surface of PSSA, which shows that the primary radical groups on the surface of separator have been changed in the presence of Li-PSSA@AA layer.

Table 1. Thickness and weight of different membrane separators.

Number	Separator	**2 wt%-coated**		**5 wt%-coated**		**8 wt%-coated**	
		Thickness (μm)	Weight (mg)	Thickness (μm)	Weight (mg)	Thickness (μm)	Weight (mg)
1	Celgard 2500	25	97.91	25	98.63	25	98.97
2	After $K_2Cr_2O_7/H_2SO_4$	23	98.29	23	98.91	23	99.34
4	After 5 wt% Li-PSSA@AA	24	102.6	24	114.8	25	117.9

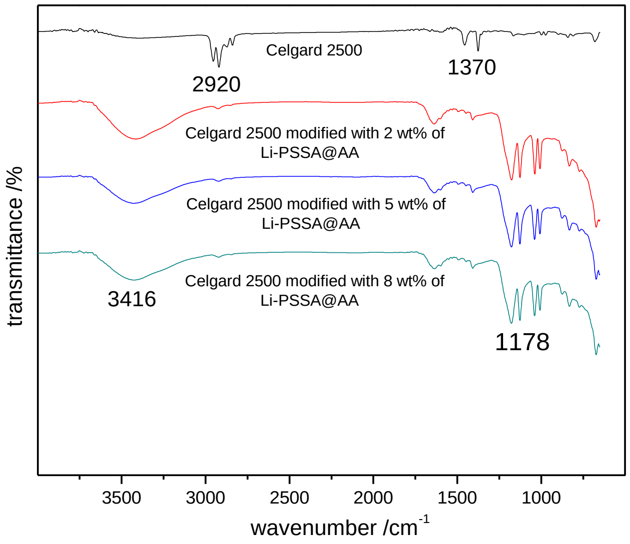


Figure 1. FTIR spectra of separators modified with different content of Li-PSSA@AA.

The SEM images of the modified separators and cathodes are depicted in Figure 2. It's shown that there are series of closely packed pores on the surface of Celgard 2500. Figures 2b and 2c indicate that for the separator coated with 2 wt% and 5 wt% Li-PSSA@AA, the pores on Celgard surface were partly filled with the polymeric ionomer. A further high concentration of Li-PSSA@AA solution (8 wt%) was used to coat Celgard as shown in Figure 2d. It can be seen that the pores on the Celgard surface were almost blocked completely. To balance Li ions transfer and sulfur suppressing, the optimized Li-PSSA@AA concentration can be selected as 5 wt.%. As shown in Figures 2e and 2f, after coated with Li-PSSA@AA, the surface of the cathode was also covered by a thin layer of the polymeric ionomer. However, it still has the distributed fissures on its surface. These fissures are important for the transfer of lithium ions and guarantee the electrochemical performance of LSB.

The contact angles of different separator's surface were showed in Figure 3. In Figure 3a, as a hydrophobic separator, the contact angle of Celgard 2500 is larger than 90°, indicating its completely hydrophobicity. This characteristic will weaken the compatibility between separator and electrolyte and therefore deteriorate the practical performance of LSB. Upon being coated with Li-PSSA@AA, the contact angle dramatically decreases to 11.06° in Figure 3b, which is much smaller than that of pristine Celgard separator. This confirms that such modification can effectively change the separator from hydrophobic to hydrophilic obviously.

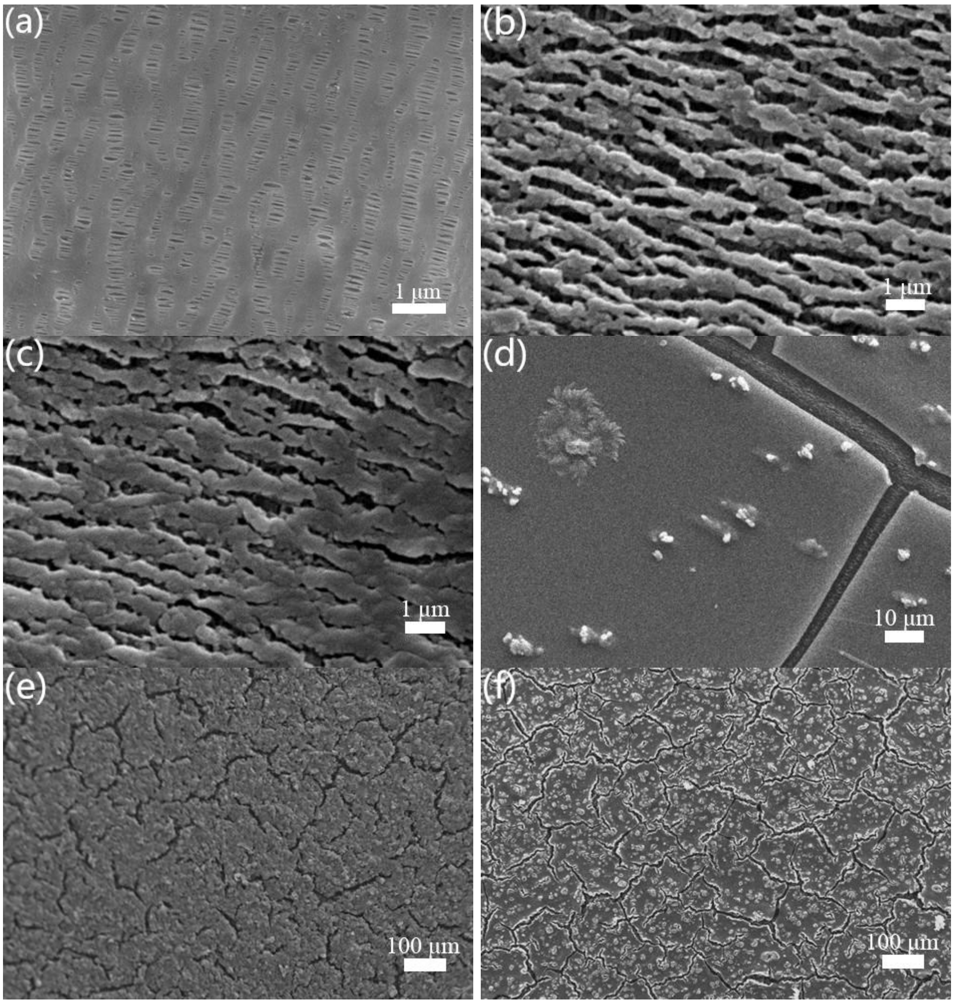


Figure 2. Morphology of different cathodes and separators: **(a)** Celgard 2500; **(b)** separator coated with 2 wt% Li-PSSA@AA solution; **(c)** separator coated with 5 wt% Li-PSSA@AA solution; **(d)** separator coated with 8 wt% Li-PSSA@AA solution; **(e)** original sulfur cathode; **(f)** sulfur cathode coated with 5 wt% Li-PSSA@AA solution.

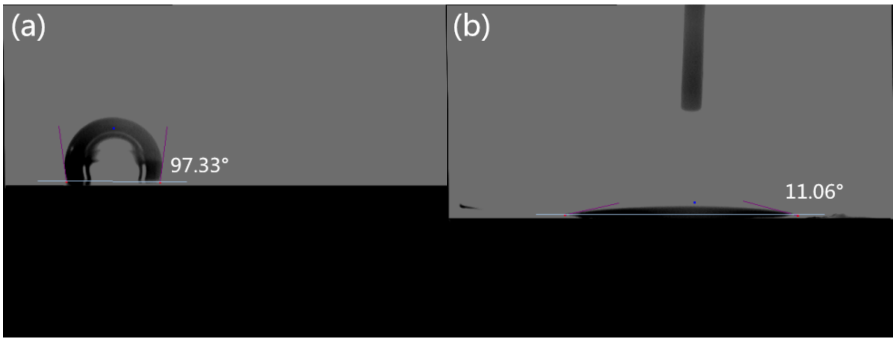


Figure 3. Contact angles with deionized water of: **(a)** Celgard 2500; **(b)** separator coated with Li-PSSA@AA solution.

To get the distribution of elements on the surface, the separators were characterized with the EDS, and the detailed result has been summarized in Table 2. It listed the atomic ratio of different elements on the surface of separators before and after the modification of Li-PSSA@AA. As shown in the table, the main elements on the surface of Celgard 2500 was C and without S element appeared. But after the modification, the surface of the composite separator was found mainly covered with element S, which was attributed to the $–SO_3^-$ on the Li-PSSA. We can see that as the concentration of Li-PSSA@AA increased, the atomic% of S element also increased. It means that the higher concentration of solution Li-PSSA@AA can lead to higher content of coating loads.

Table 2. EDS data (atomic%) of different membrane separators.

Number	Separator	C	O	S
1	Celgard 2500	98.32%	1.68%	0
3	Composite separator with Li-PSSA@AA of 2 wt%	63.46%	31.83%	4.45%
4	Composite separator with Li-PSSA@AA of 5 wt%	68.39%	26.62%	4.99%
5	Composite separator with Li-PSSA@AA of 8 wt%	64.56%	29.28%	6.16%

3.2 Electrochemical performance

Figure 4 depicts EIS profiles of different separators used in this work. As shown in Figure 4a, the impedance of modified separators are slightly larger than that of Celgard 2500 because the Li-PSSA@AA introduced can increase the obstruction of separator for the ions. As shown in Figure 4b, however, the Nyquist plots of all the fresh batteries using modified separator display a single semi-circle in higher (the left side) frequency region, and an inclined line in lower (the right side) frequency region. The semi-circle in high-frequency region is attributed to the total resistances of the electrolyte interface formed on electrode surface (R_S) [17, 18], including the electrode and electrolyte resistances. The inclined line in low frequency region corresponds to Warburg impedance (W_o) for a semi-infinite Warburg diffusion process [19, 20]. By comparing the value of Rs in different batteries, it can be concluded that the introduction of low concentration (2 wt%) Li-PSSA@AA decreases slightly the impedance of battery. Nevertheless, the impedance decreases in case that high concentration (8 wt%) used. Because of the changes in the polarity of separator surface, the compatibility between separator and electrolyte is greatly improved to promote the electrochemical reaction in LSB and finally reduce the impedance. The EIS results indicate that the Li-PSSA@AA could be used as a promising additive to modify the surface of separator.

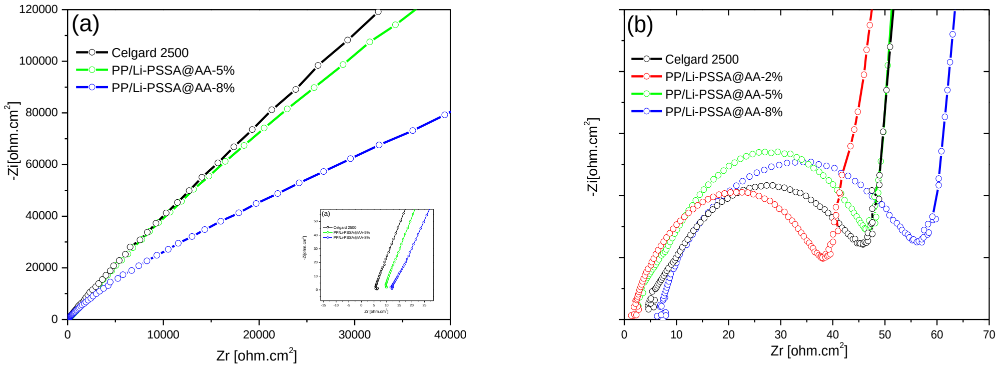


Figure 4. EIS plots of **(a)** different separators before and after coated with Li-PSSA@AA; **(b)** fresh batteries using different separators.

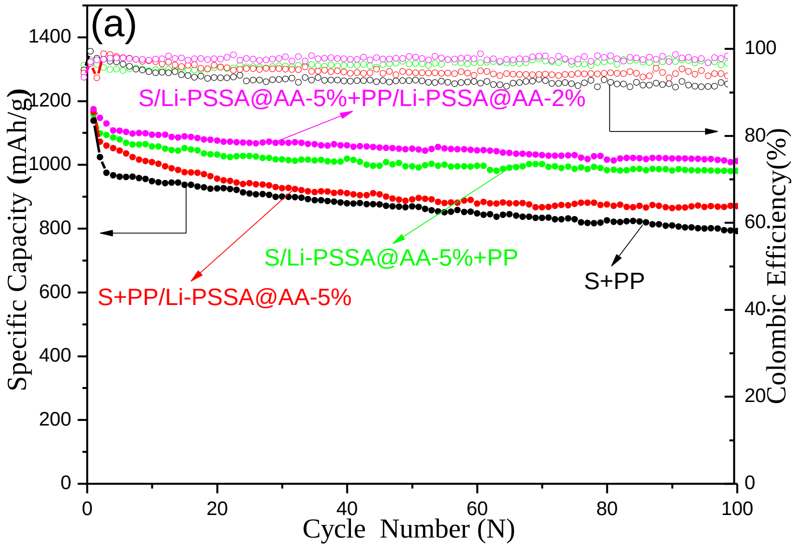
The cyclic discharge performance of LSBs assembled with different cathodes and separators between 1.5 V and 2.8V at room temperature is shown in Figure 5. As depicted in Figure 5a, at the same current rate of 0.1 C (based on the weight of sulfur, 1C=1675mA/g), the battery assembled with modified cathode and pristine Celgard 2500 had a similar initial specific capacity (S-5%Li-PSSA/AA@PP, 1157 mAh/g) with that of cell without any modification (S@PP, 1138 mAh/g), as well as those assembled with pure cathode and modified separator (S@PP-5%Li-PSSA/AA, 1165 mAh/g). This is due to that these batteries were assembled under same condition at the same time. However, the battery with modified cathode kept a specific capacity of 980 mAh/g after 100 cycles, demonstrating the best cycling stability with the highest capacity retention of 84.70%. The battery without modification exhibited a fastest decay of specific capacity, finally getting a retention of 69.42% (790 mAh/g) after 100 cycles with a decay rate of 0.306% per cycle, which is much higher than 0.153% of the cathode modified battery. The improved battery performance is believed to be resulted from the obstacle formed by Li-PSSA that can effectively trap polysulfide through electrostatic repulsion. The $-SO_3^-$ groups on PSSA has a strong electrostatic force to prevent the polysulfide to pass, but the Li^+ was permitted to transfer freely at the same time [21-23]. Through restricting the polysulfide within the space between cathode and separator, the shuttle effect and the loss of active material during charge-discharge process can be greatly reduced, and finally the cyclic stability of the battery was improved.

By comparing the battery performance with modified cathode and separator, it should be noted that the modified cathode can bring us higher capacity retention, because the Li-PSSA@AA layer on cathode surface can effectively trap polysulfide and avoid them dissolving in electrolyte. Nevertheless, the Li-PSSA@AA layer on separator can only trap them after dissolved in electrolyte. In this sense, the modified cathode can afford a greater improvement in electrochemical performance compared to the modified separator. Moreover, when the LSB was assembled with both modified cathode and modified separator, the battery behaved an excellent electrochemical performance with an initial specific capacity of 1174.5 mAh/g and a capacity retention of 85.82% or a decay rate of 0.142% per cycle after 100 cycles. So we can summarize that the double modification both in cathode and separator can bring us the best improvement of cyclic performance for lithium-sulfur battery.

The cyclic performance of LSBs with different cathodes was further conducted at the current rate of 0.1 C and 0.5 C. As shown in Figure 5b, during the first 10 cycles at 0.1C, the capacity of all the batteries decreased quickly and then levelled off after 20 cycles. It is apparent that the LBS modified with 5 wt% Li-PSSA@AA showed the best cyclic performance compared to others. Both modified cathode and separator in LSB showed effectively suppressing of polysulfide and provided a short cut paths for lithium ions. The cyclic performances at 0.5C in Figure 5c were similar with those at 0.1C.

The rate performance using the cathodes modified with different Li-PSSA@AA concentration was also investigated at various current densities and C-rates from 0.1C to 5C. As shown in Figure 5d, during the first 5 cycles, the cells were operated at 0.1C rate and then the current rate changed to 0.2C, 0.5C, 1C, 2C, 5C in turn. Finally the charge/discharge rate was changed back to 0.1C. At the rate of 0.1C and 0.2C, the capacity of cathode modified battery using 8 wt% Li-PSSA@AA was higher than others, however, the capacity decayed quickly and showed an even worse capacity when the rate changed back to 0.5C or higher, comparing to those using 2 wt% and 5 wt% Li-PSSA@AA.

Presumably, this phenomenon is attributed to the extra thickness layer of Li-PSSA@AA coated on the surface, leading to the path block for lithium ions. On the other hand, when the charge/discharge rate was decreased to 0.1C after continuous 45 cycles, the capacity using 5 wt% Li-PSSA@AA almost recovered to its origin value.



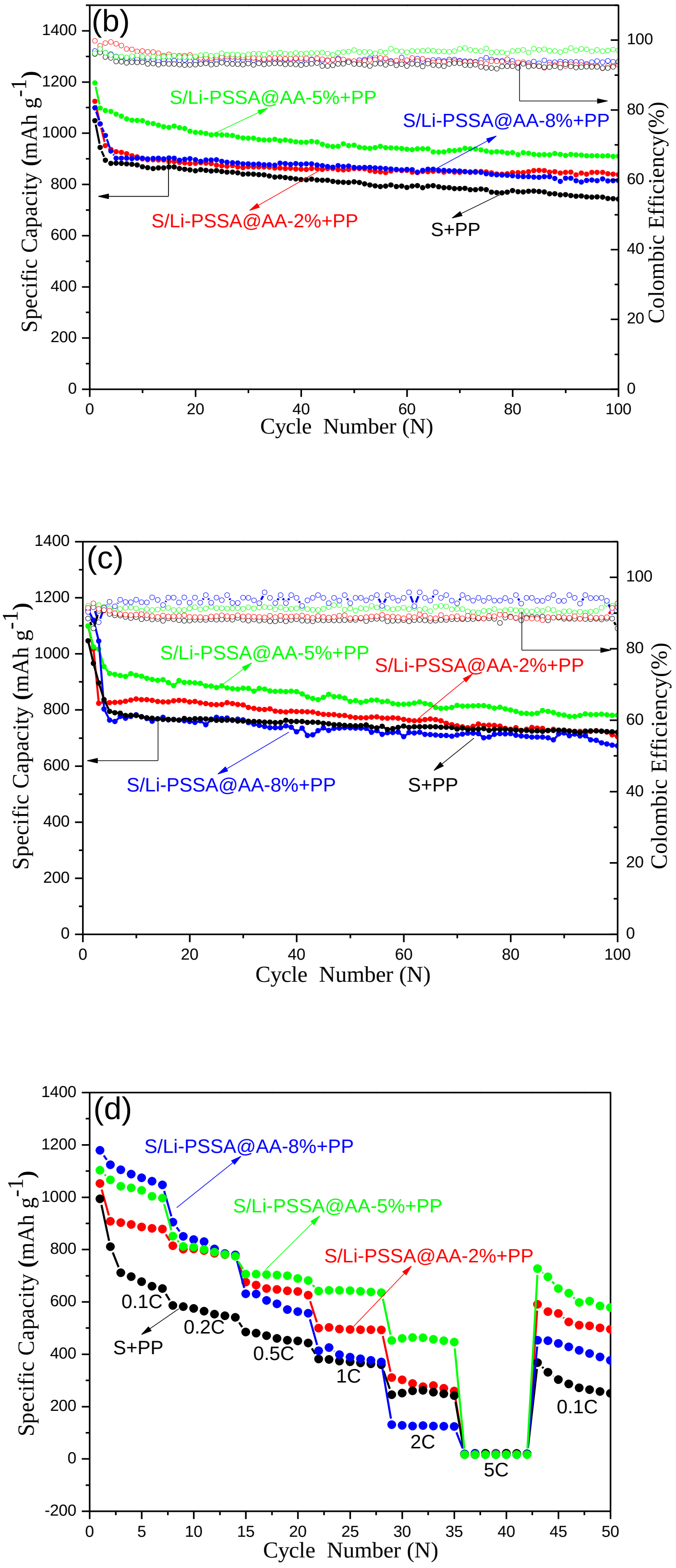


Figure 5. Discharge cyclic performance of LSB: **(a)** using different cathodes and separators at 0.1C; **(b)** using different cathodes at 0.1C; **(c)** using different cathodes at 0.5C; **(d)** using different cathodes at various current rates.

This result also shows that the 5 wt% Li-PSSA@AA is the best solution concentration for both cathode and separator modification.

The excellent cyclic stability and rate performance of the modified lithium-sulfur batteries are believed to be resulted from the barrier formed by Li-PSSA with proper concentration, which served as the obstacle between cathode and separator to trap polysulfide and avoid it dissolve in the electrolyte. In the charge-discharge process, this barrier could reduce the shuttle effect of polysulfide and the loss of active material. Some similar modification including polymers has been applied to modify the LSB[24-28]. Through introducing the functional coating, the purpose of suppressing the shuttle effect has been reached[29-31], and the electrochemical performance of these composite cathodes and separators for LSB were summarized in table 3.

Table 3. Different modification for lithium-sulfur battery and their electrochemical performance.

Modification	Initial capacity (mAh/g)	Retention rate	Decay rate (per cycle)	Ref.
Oxidized multiwall carbon nanotube	1174.5 at 0.1 C	85.82% (100 cycles)	0.142%	This work
Graphene oxide	920.0 at 0.1C	77.00% (100 cycles)	0.230%	[24]
Nafion	1084.0 at 0.2C	81.09% (100 cycles)	0.189%	[25]
N-doped mesoporous carbon	1364.0 at 0.2 C	76.25% (100 cycles)	0.238%	[26]
MPC/PEG	1060.0 at 0.2 C	51.03% (75 cycles)	0.653%	[27]
MWCNT	1324.0 at 0.2C	66.54% (150 cycles)	0.335%	[28]

From these typical works in Table 3, we can know that the modification of Li-PSSA@AA for improving the cyclic performance of LSB is rational. This work shows a high retention rate after 100cycles at 0.1 C. The lithium-sulfur battery assembled with modified cathode and separator exhibited excellent electrochemical performance, which was because that the introduction of Li-PSSA@AA could trap the polysulfide, reduce the shuttle effect of polysulfide and the loss of active materials. As a result, excellent cyclic stability and rate performance can be obtained for the lithium-sulfur battery.

4. CONCLUSIONS

According to the experiment results taken for various lithium-sulfur batteries, the modification on both separator and cathode using Li-PSSA@AA solution can effectively improve the electrochemical performance of LSBs. The unique combination of Li-PSSA and AA can substantially decrease the shuttle effect of polysulfide into electrolyte during the charge/discharge process, and improve the cyclic stability of LSBs. The as-assembled batteries showed a significant improvement in their performances over pristine battery. Particularly, the LSBs assembled using both modified cathode and modified separator exhibited a high specific retention of 85.82% after 100 cycles at 0.1C, which is obviously superior to the traditional battery without any modification. Therefore, the modification of both separator and cathode surface using Li-PSSA@AA solution was proved to be a potential strategy for LSB’s application.

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