



Research Article

# Effects of gelation behavior of PPC-based electrolyte on electrochemical performance of solid state lithium battery

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## Abstract

Polypropylene carbonate (PPC)-based solid state electrolyte was fabricated by using a cellulose membrane as a skeleton. The gelation behavior of the PPC-based solid electrolytes in solid-state lithium batteries was found, and the effect of this behavior on battery performance was studied. It was found that the solute lithium salt in the matrix greatly promoted the gelation of the PPC-based solid electrolyte under heating conditions upon contact with metallic lithium. This behavior allows the room temperature conductivity of the electrolyte to be directly increased by two orders of magnitude, on the order of  $10^{-3}$  S/cm, and also greatly improves the wettability of the electrode interface. The mechanism of in situ gelation allows the solid state battery to actually operate in a gel state. Since the actual electrochemical window of the electrolyte is only 3.8 V due to gelation, the electrolyte membrane continuously undergoes side reactions during the high voltage cycle, resulting in a continuous decrease in cycle efficiency.

**Keywords** PPC-based electrolyte · Gelation behavior · Solid state battery · Ionic conductivity

## 1 Introduction

Solid electrolyte (SE) is the core of research in all-solid-state battery technology. The current mainstream SE includes the solid inorganic electrolyte and the solid polymer electrolyte (SPE). The former is mainly divided into two major directions of oxides and sulfides; the latter mainly includes polyethylene oxide (PEO), polycarbonate (PC) and the like. For SPE, although the RT ionic conductivity and stability are not as good as inorganic substances, it has great advantages in comprehensive electrochemical performance and preparation, such as: good interface wetting, flexible film preparation process, acceptable ion migration number and electrochemical window [1–9]. In fact, good interfacial wetting and simple thin film process are unique advantages of SPE [10, 11].

Compared with the most studied PEO solid electrolytes [8], PC-based solid electrolytes have obvious advantages

[6, 7, 12–19]: (1) RT conductivity can reach  $10^{-4}$  S  $\text{cm}^{-1}$  or more; (2) The electrochemical window can reach more than 4 V; (3) The ion migration number can reach 0.5 or more. These advantages are the focus of the electrochemical performance of SPE. Cui made a comprehensive study on PC-based solid electrolytes, and proposed the concept of “rigid and flexible” to improve the mechanical strength of polymer matrix and obtained all solid state cell with long cycle stability [6, 13, 18]. Tominaga obtained a solid electrolyte membrane with the electrochemical window of 5 V and ion migration number of more than 0.5 by studying the polyethylene carbonate (PEC) solid electrolyte, and the RT conductivity reached  $10^{-5}$  S  $\text{cm}^{-1}$  at a lithium salt addition of 188 mol%, metal lithium cell matching  $\text{LiFePO}_4$  positive electrode had excellent rate performance [20]. All of the above work shows that PC-based SPEs have great application prospects.

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What’s the main reason for PC-based SPEs have so many excellent RT characteristics different from conventional SPEs? In our work, we prepared PPC-based solid electrolyte and studied the reaction process in solid state battery, found that a gelation process could be occurred when PPC was in contact with metallic lithium, which greatly influenced the electrochemical performances of the solid state battery.

### 1.1 Material preparation

In order to improve the mechanical strength of the PPC solid film, a commercial cellulose membrane was used as a support skeleton for the PPC solid film [13, 18, 19]. The entire preparation process was shown in Fig. 1: First, 5 g PPC was sufficiently dissolved with 10 g N,N-dimethylformamide (DMF), followed by addition of a certain amount of LITFSI, and then the mixture was thoroughly stirred to obtain a premix. A 100 μm thick premix was scraped on the steel plate, then a porous cellulose film was laid, and a slurry having a thickness of 400 μm was scraped off, finally dried in a vacuum oven at 100 °C for 24 h, peeled off to obtain the PPC-based SPE supported by a skeleton (CPPC-SPE). The similar preparation process was used to prepare a PPC-based SPE without skeleton (PPC-SPE) and without lithium salt (PPC). The thickness of the electrolyte membrane prepared above was 100 ± 5 μm.

## 2 Electrochemical performance

### 2.1 RT conductivity of dry film

Unlike the PEO, PPC is a glassy state with high hardness at room temperature [14, 24]. so if simply using the conventional method, assembling the of “SS (stainless steel)/SPE/SS” structure (SS symmetrical cell) blocking ion migration for measuring the impedance of the SPE may cause the

testing error of the SPE to be excessive due to the poor contact of the SS/membrane interface [6, 7, 21]. Therefore, in this experiment, the “soldering” of the SS/membrane interface was carried out using a polar solvent, so that the interface contact of the blocking electrode was greatly improved, and the conductivity obtained by the test was in accordance with expectations [6, 14, 15]. It can be seen from the Fig. 2, that the RT conductivity of the CPPC-SPE reaches the maximum, which is  $4.3 \times 10^{-5} \text{ S cm}^{-1}$  when the amount of lithium salt added is 30%. So we choose 30% as the lithium salt content of the following SPE unless otherwise specified.

### 2.2 Ion migration number and electrochemical window

A metal lithium symmetrical cell “Li/CPPC-SPE/Li” was assembled to directly test the ion migration number of the CPPC-SPE. However, it was found during the

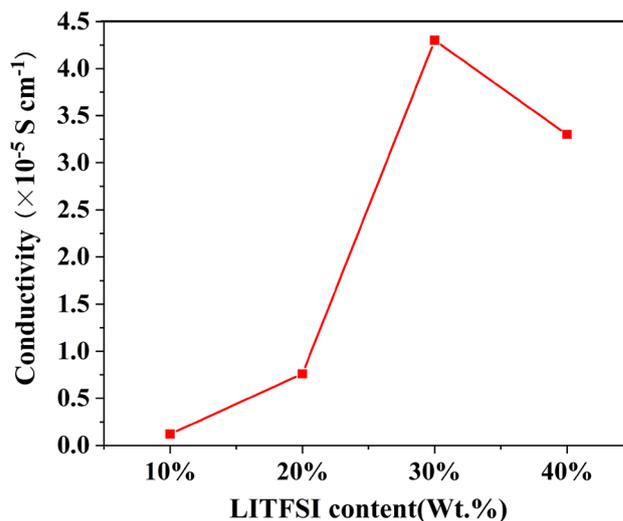
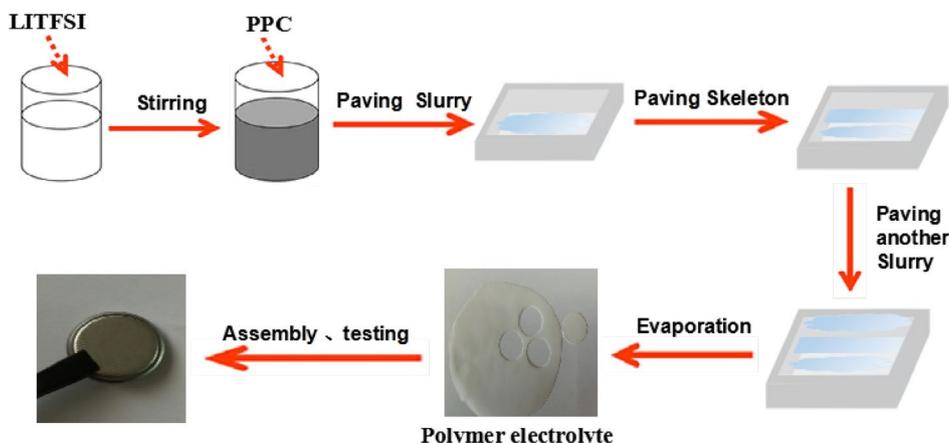


Fig. 2 Ionic conductivity of CPPC-SPE in RT before gelation

Fig. 1 Preparation process of PPC-based solid electrolyte membrane



test that the open circuit voltage (OCV) of the cell was extremely unstable and the DC bias could not be applied correctly. We compared the OCV of the “SS/CPPC-SPE/Li” (called “LSV cell”) and found the same phenomenon, as shown in the Fig. 3b, it is also impossible to test its electrochemical window [6, 7, 9, 16, 20]. Corresponding to the SS symmetrical cell that was not processed before, the unstable OCV should be caused by the poor interface contact.

In order to prove the conjecture above, all the cells at 100 °C for 12 h before testing. After that, we found that the OCV of the symmetrical cells are stable other than “SS symmetrical cell”. The impedance spectrum and polarization curve obtained by the test are shown in the Fig. 3c. It has been calculated that the ion migration number is 0.49, which is more than twice that of PEO-SPE [8]. The polarization curve of the LSV cell was successfully obtained, shown in the Fig. 3d. The electrochemical window of CPPC-SPE reached to 4.6 V, which was close to the literature [6, 13].

So what happened inside PPC-based SPE cell during heating so as to get the normal results? We conducted follow-up experiments.

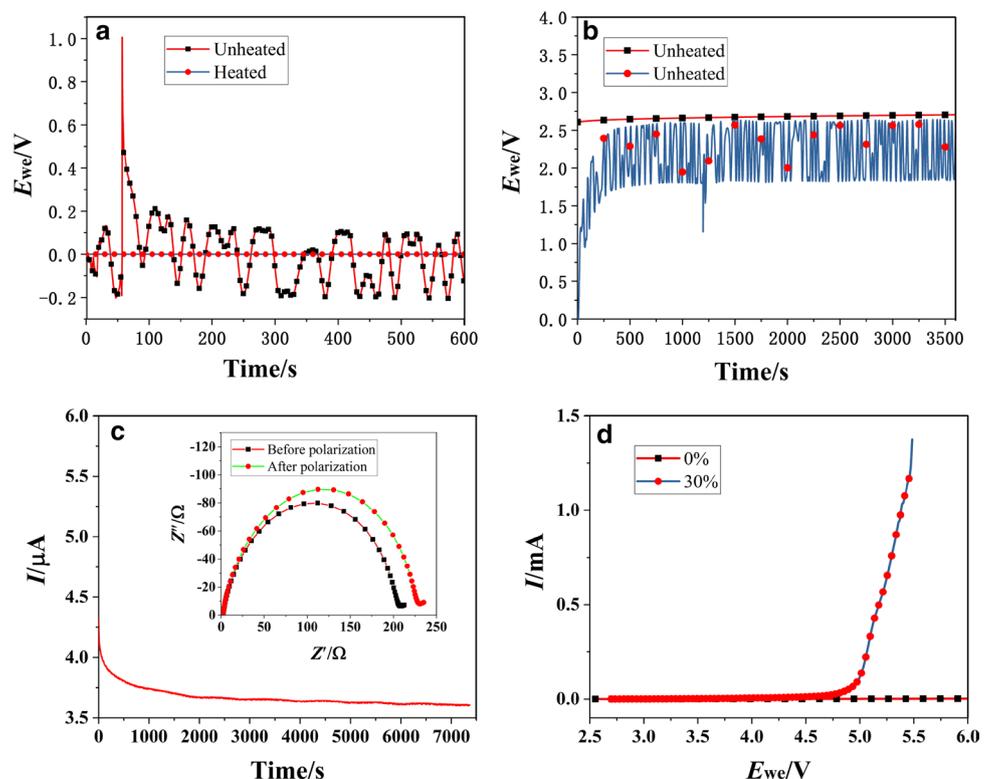
### 3 Gelation of CPPC-SPE

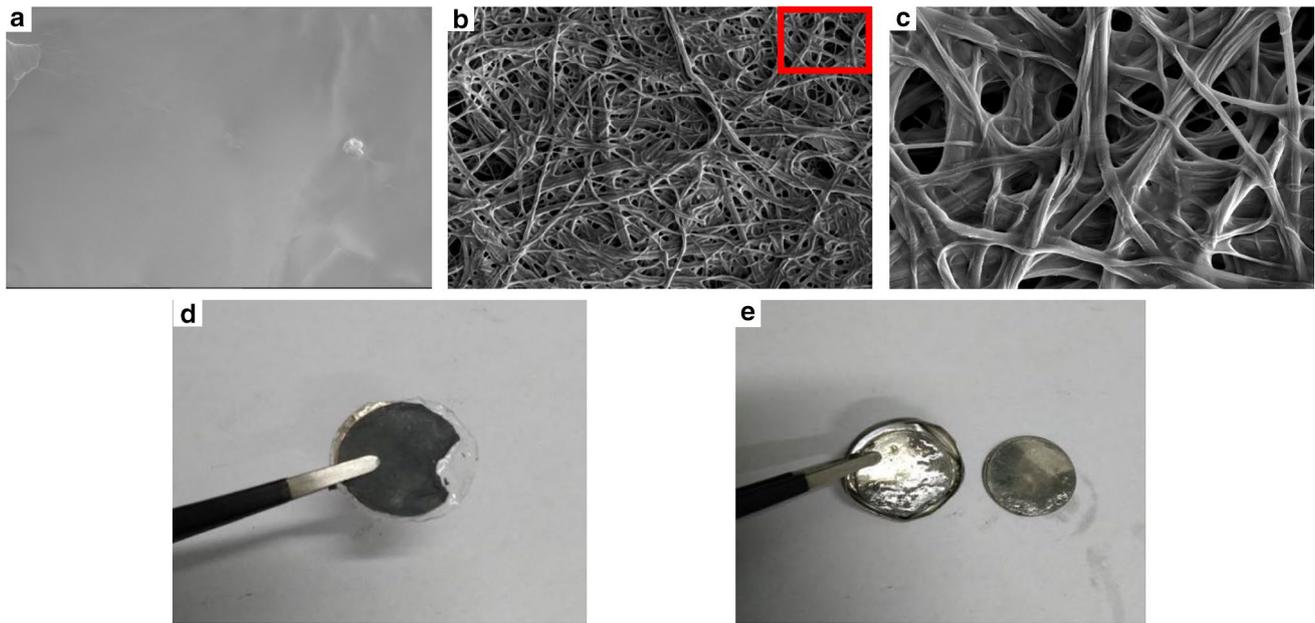
#### 3.1 Scanning electron micrograph

A lithium symmetrical cell was assembled with CPPC-SPE to observe the change of CPPC-SPE in contact with metallic lithium under different conditions. The surface of the CPPC-SPE not in contact with metallic lithium is shown in Fig. 4a, and the surface is smooth. However, after heating, as is shown in Fig. 4b, the surface skeleton of the film is exposed to a serious extent, and only a small part of the voids are filled with a certain amount of polymer along with some obvious liquid. It demonstrates that CPPC-SPE will gel after heated when in contact with the metal lithium [21]. To further illustrate the problem, PPC-SPE under the same conditions was applied, and it was found that the membrane “disappears” after heating, in the meanwhile, a certain amount of liquid generated while the membrane still existed and hardly changed in the unheated one.

Both two experiments can show the fact that: (1) Heating can lead to gelation of the CPPC-SPE; (2) when there is no backbone support, the polymer will completely liquefy. Therefore, the skeleton plays an important role in the PPC-based solid metal lithium cell. So how does the gelation occur during the heating process?

**Fig. 3** **a** OCV voltage of the unheated lithium symmetrical cell; **b** OCV voltage of the LSV cell; **c** polarization curve and corresponding impedance spectrum of the lithium symmetrical cell after heating; **d** step voltage-current curve of LSV cell





**Fig. 4** **a** Unheated film surface photo  $\times 2000$ ; **b** heated SEM photo  $\times 2000$ ; **c** partially magnified SEM photograph  $\times 10,000$  in red frame of **b**; **d** digital photo of pure PPC after heated; **e** digital photo of PPC-SPE after heated

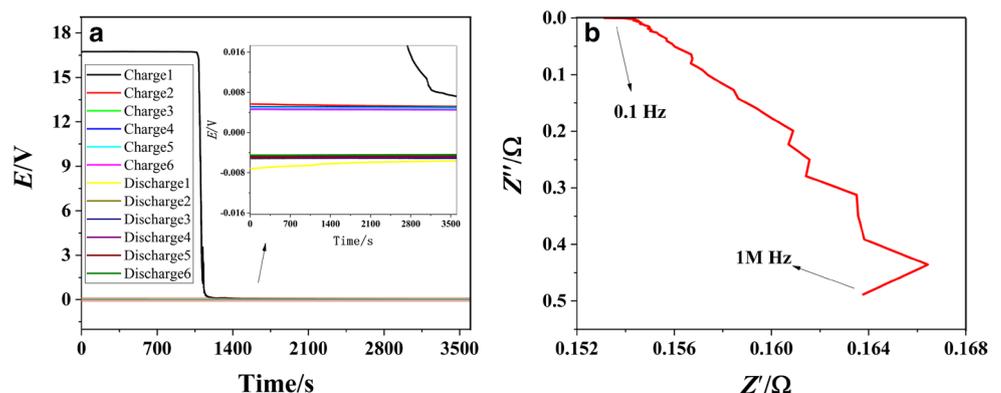
### 3.2 Short circuit test

In order to characterize the detail of the CPPC-SPE gelation process during heating, lithium symmetrical cell assembled with PPC-SPE was applied. Electrochemical workstation was used to perform constant current charge and discharge at  $100\text{ }^{\circ}\text{C}$  to observe the polarization voltage. It was charged—1 h followed by discharged—1 h with the current of  $0.5\text{ mA}$ . The polarization voltage reflected the change of the PPC-SPE inside cell.

At the beginning of the first charge, the polarization voltage was as high as  $16.8\text{ V}$ , which made it possible to calculate an interface impedance of up to  $16,750\text{ }\Omega$  [22–24]. When  $1085\text{ s}$ , the polarization voltage suddenly dropped. It cost  $47\text{ s}$  from  $16.8\text{ V}$  to  $1\text{ V}$ , then dropped to  $8\text{ mV}$  after  $2028\text{ s}$ , and the interface impedance was

only  $6\text{ }\Omega$ , the cell being circuit obviously. The interface impedance varied from  $16,750\text{ }\Omega$  to  $6\text{ }\Omega$ , indicating that the polymer matrix had begun to gel rapidly during the time, which greatly improved the wettability of the electrode interface. And after quickly dropped, the polarization voltage was slowly close to  $0\text{ mV}$  whatever charge or discharge due to the slowly increasing wettability of the interface. After the test completed, the cell impedance was further tested. The impedance spectrum shown in the Fig. 5b also proves the cell short-circuited. In combination with the state of gelation of the heated PPC film concluded from the change of polarization voltage, it can be inferred that in the operation of the circulating cell, if there is no skeleton as the separation layer, it is likely to be thoroughly short-circuited after the cell is heated about  $3160\text{ s}$ .

**Fig. 5** **a** Constant current charge and discharge curve; **b** impedance spectrum after charge and discharge



The fact indicates that the cellulose film skeleton is necessary, not only to improve the mechanical strength, more importantly for physically separating, that is, when the PPC gels to “disappear”, it acts as a separator to prevent short circuit [21].

## 4 The cause of gelation

### 4.1 Analysis of the cause of PPC gelation

In order to find out the cause of PPC gelation, we assembled a cell with different electrode matching, and the structure was “electrode 1/CPPC-SPE/electrode 2”. As shown in Table 1, under different conditions, we used “Yes” or “No” to indicate whether the corresponding phenomenon occurred.

The phenomenons of the above control experiments show that: (1) only when exposed to metallic lithium, the film can appear liquid; (2) if the SPE is dry, the impedance spectrum cannot be obtained, indicating that the interface impedance is extremely large or unstable; (3) the addition of lithium salt and heating is necessary for ensuring the liquid generates when SPE is in contact with the metallic lithium. In fact, the liquid also appears when CPPC-SPE in contact with Li(OH)<sub>2</sub> [25], indicating that the trace of Li(OH)<sub>2</sub> impurity on the surface of the metallic lithium also causes CPPC-SPE gelation [21].

Based on the above analysis, we can make the following speculation: the reason that the metal lithium solid state cell assembled by the PPC-based electrolyte can circulate well is the positive and negative electrodes infiltrated well due to the gelation of PPC. It also indicates that probably the PPC-based solid electrolyte membrane is not suitable for graphite or silicon carbon anode because of high interface impedance without gelation.

### 4.2 PPC gelation product analysis

In order to determine the product after PPC degradation, we used Fourier transform infrared spectroscopy to analyze the characteristic groups of the electrolyte membrane

under different conditions. The results were shown in Fig. 6. Lithium ions form complexes with ester groups of PPC [13]. The anion and anion of lithium salts exist in the form of free ions, ion pairs and aggregates. These existences make the segment and chemical bond of PPC unstable. Under heating conditions, the segmental structure of PPC gels to form small molecular segments [21].

Comparing the 30% lithium salt CPPC-SPE before and after heating, it is found that the characteristic spectrum of PC appeared in the heated state. Heating promotes the breakage of the ester group and produces PC, and a small amount of hydroxyl-containing segments. It is likely that the heating made the kinetic conditions better in the corresponding environment.

Comparing the lithium-free salt CPPC-SPE before and after heating, it is found that after heating, only a wide and weak hydroxyl absorption band appears in the range of 3100–3700 cm<sup>-1</sup>, which indicates that the surface of the pure PPC film still forms a hydroxyl-based complex after heated, but there is no characteristic band of PC, indicating that the reaction between PPC and lithium sheet does

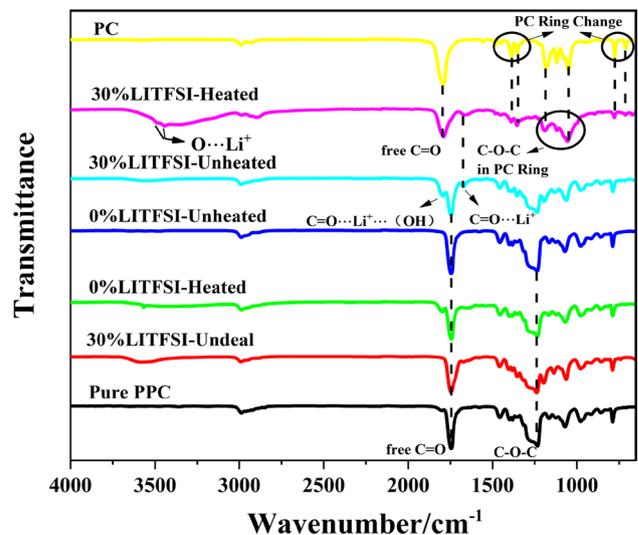


Fig. 6 Fourier infrared spectroscopy of CPPC-SPE under different conditions

Table 1 Electrolyte film state under different electrode matching

LITFSI content	30% LITFSI				Free Lithium symmetrical					
	Type of cell									
Phenomenon	NCM523 symmetrical	SS symmetrical	Lithium symmetrical	LSV Cell						
Heated or not?	√ (Yes)	× (No)	√	×	√	×	√	×	√	×
Dry or not?	×	√	√	√	×	√	×	√	√	√
Can getting impedance spectrum?	×	×	×	×	√	×	√	×	×	×

not produce PC, and the corresponding SPE is still in a dry state. Similar to the transient complex formed by PPC and lithium ion, a complex of carbonyl and lithium ion is formed on the left side of the carbonyl group at  $1800\text{ cm}^{-1}$ , and this difference is likely to be caused by little dissolution of lithium hydroxide in the polymer matrix. The reason that hydroxyl appeared is the decomposition of PPC in alkaline environment to produce hydroxyl segments.

In the comparison CPPC-SPE of 30% and lithium-free salt, it can be seen that in the high wavelength region, the band positions are almost identical, but there is a difference in intensity. Hydroxyl and hydroxy-lithium are concentrated in this region. Combined with the mechanism of PPC gelation proposed by Cui [21] as shown in Fig. 7, it can be found that the lithium hydride complex formed by the small molecular segments after the preliminary gelation of PPC is essential, and the complex is unstable, further reaction under the catalysis of metallic lithium produces monomeric PC and smaller segments. It is apparent that the addition of the electrolyte lithium salt is very advantageous for the formation of such a complex of hydroxy-lithium for accelerating the gelation of PPC. In addition to this, we have also found the O-Li<sup>+</sup> chemical bond obtained by replacing the alcoholic hydroxyl group with lithium ions after heating the lithium cell.

### 5 RT conductivity of gelled film

PPC-based SPE is all solid when assembled, but in a gel state while actually operates, which is a major feature of PPC-based SPE. Therefore, it is more practical to

determine the room temperature ionic conductivity after PPC gelation.

It can be seen from the Fig. 8 that the gel state electrolyte membrane of CPPC-SPE can reach the order of  $10^{-3}\text{ S/cm}$  at room temperature [1], which is equivalent to the liquid electrolyte. Compared to the ungelatinized PPC film, the RT conductivity increases by nearly two orders of magnitude, which is very beneficial for cell performance.

As be seen from the Fig. 8, the RT conductivity of the gel electrolyte membrane of 10% lithium salt and 30% lithium salt is only double the gap, which is almost same to show in non-power devices in performance [1]. However, for the process performance, since the organic lithium salt is

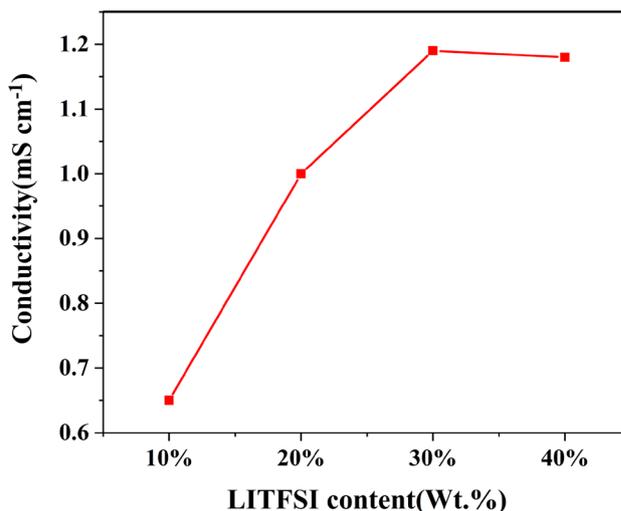


Fig. 8 Ionic conductivity of CPPC-SPE in RT after gelation

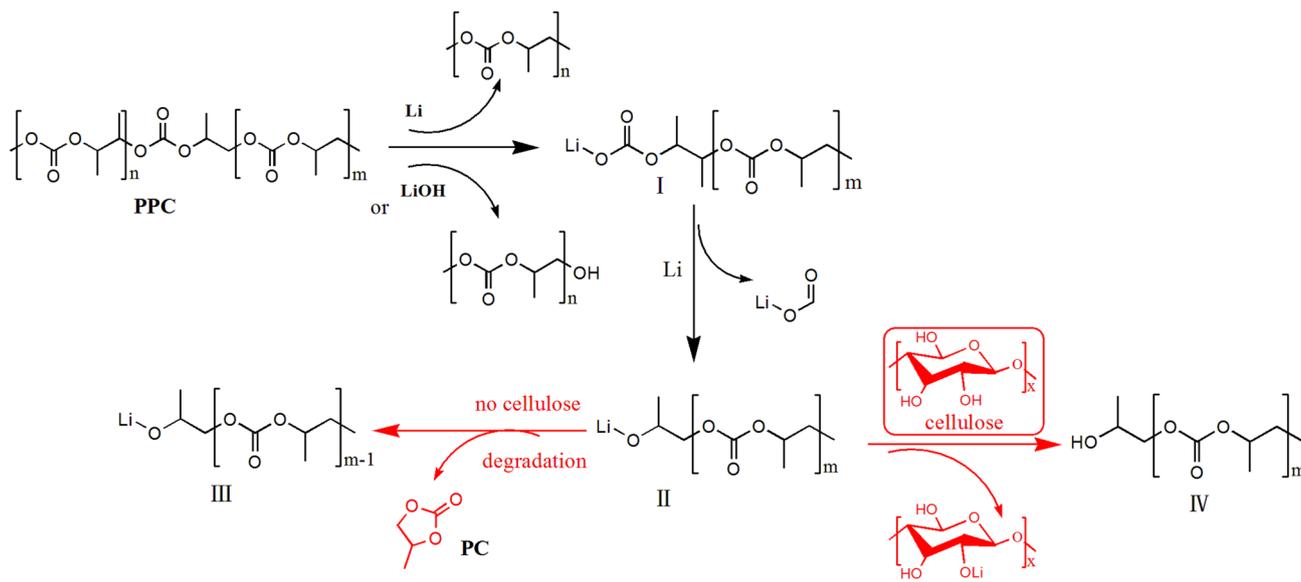


Fig. 7 Gelation mechanism of PPC [21]

highly viscous, the electrolyte membrane becomes wet and sticky, and this characteristic is remarkable at a high lithium salt concentration, which is disadvantageous for the scale production of the PPC-based SPE. Therefore, considering the comprehensive consideration, it is recommended to use a low concentration of lithium salt.

## 6 Cell performance

The above-prepared CPPC-SPE with a LITFSI content of 10 wt% assembled 2025 button cell matching positive electrode of NCM523 were tested for cycle performance. The charge and discharge system was: “constant current (CC) charge–constant voltage (CV) charge–constant current discharge”, the voltage interval was 2.8–4.3 V.

As can be seen from the Fig. 9a, the capacity–voltage curve of the cell is the same as that of the conventional liquid cell [26]. However, the cell's first cycle efficiency is lower, mainly because the cell has been overcharged in the first cycle. This is because there is a electron exchange reaction, the oxidation state ions of the transition metal oxide are reduced by PPC on the cathode reported by Cui [27], thus causing a fake high charge capacity. During the entire charge and discharge process, The proportion of capacity occupied by CV charging gets higher and higher, and the amplitude of the rise is increasing, eventually exceeding the charging capacity of the CC stage. When

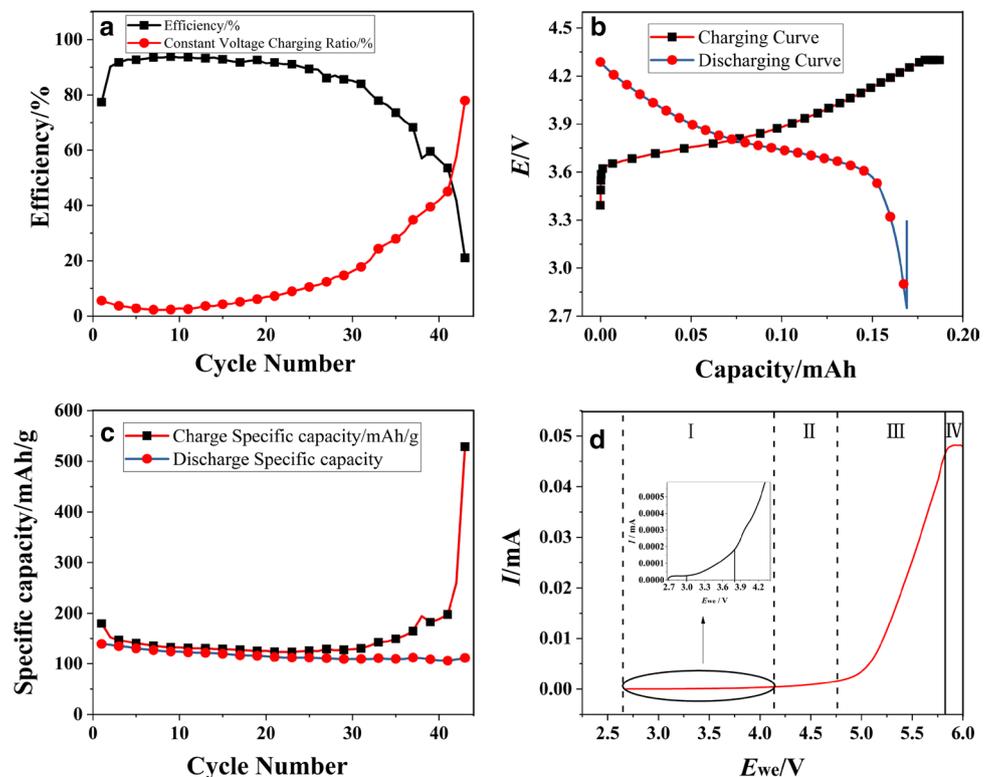
the ratio of the CV charging capacity starts to rise, the discharge capacity does not increase significantly, obviously, which indicates that the charging capacity in the CV is not all used for the deintercalation of lithium ions from the positive material, but other side reactions have occurred.

Comparing the electrochemical window obtained by the test, it can be found that although the oxidation current of the electrolyte membrane is significantly increased at 4.6 V, However, it can be seen that after amplification, the change trend of the oxidation current has increased at 3.8 V, and further increases at 4.3 V. Therefore, we have reason to suspect that the optimum cycle voltage of the CPPC-SPE should be 3.8 V at the highest, and the higher voltage cycle will cause continuous destruction of the electrolyte membrane, which will greatly reduce the cycle performance. Obviously, it is the gelation of PPC that causes the gelled product to not withstand a high voltage of 3.8 V or more.

## 7 Conclusions

Metallic lithium can degrade PPC. When lithium is dissolved in the matrix, heating greatly promotes this process, producing PC and hydroxyl-containing short chains, which is the in situ gelation of PPC-based SPE in lithium metal cell. The liquid-containing product generated by this mechanism will greatly improve the wettability of SPE to

**Fig. 9** **a** Curve of efficiency/capacity; **b** curve of voltage–CV charging capacity; **c** charge–discharge capacity cycle curve; **d** curve of electrochemical window



the positive and negative electrodes, which is beneficial to the performance of the battery. Anyway, in situ gelation, as an important feature of PPC-based solid electrolytes, will certainly have a certain position in the development of all-solid-state cell technology.

**Funding** This study was funded by National Natural Science Foundation of China (Grant No. 51474113).

### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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