



Short communication

Stable all-solid-state potassium battery operating at room temperature with a composite polymer electrolyte and a sustainable organic cathode



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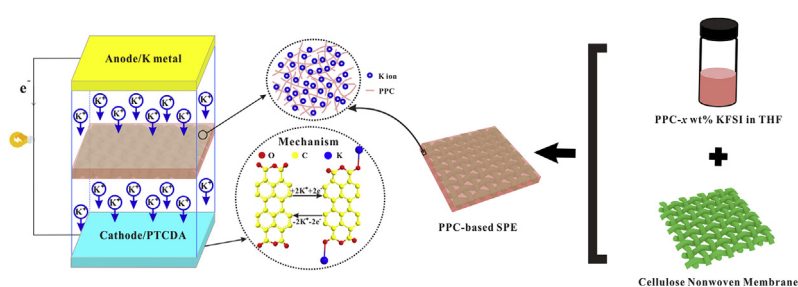
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HIGHLIGHTS

- All-solid-state potassium organic battery is assembled for the first time.
- A PPC-KFSI electrolyte with cellulose backbone SPE is firstly synthesized.
- Affection of salt concentration and temperature on SPE is investigated.
- The electrochemical performance of PTCDA battery is significantly improved.

GRAPHICAL ABSTRACT



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ABSTRACT

The safety issues of potassium battery with nonaqueous electrolytes restrict its practical applications. On the other side, the active material dissolution of organic electrodes deteriorates the performance of organic batteries. As an alternative, all-solid-state potassium batteries comprising the cathode materials of organic compounds can solve the mentioned problems. We herein succeed in demonstrating an all-solid-state potassium battery based on 3,4,9,10-perylene-tetracarboxylicacid-dianhydride (PTCDA) and solid polymer electrolyte(SPE) for the first time. The Poly (propylene carbonate) (PPC)-KFSI with cellulose nonwoven backbone SPE (PPCB-SPE) delivers an ionic conductivity of $1.36 \times 10^{-5} \text{ S cm}^{-1}$ at 20 °C. Furthermore, PTCDA cathode in all-solid-state battery provides an average discharge voltage of 2.3 V, a high initial capacity of 118 mA h g^{-1} at 10 mA g^{-1} and a stable cycling performance, showing favorable expectation for the commercial application.

1. Introduction

Lithium-ion batteries (LIBs) have gained great success in electric vehicles and powering portable devices in the last three decades [1–4]. With the ever-increasing demand in the usage of LIBs, relying on LIBs is becoming more and more uneconomical and impractical due to the rarity and uneven distribution of lithium resources [5]. Thus, it is crucial to pursue alternatives to LIBs. Two near-neighbor elements (Na

and Mg) have attracted great attention during the last two decades [6,7]. Despite the significant attention to Na-ion battery(NIB), it seems that only a few investigations have focused on potassium-ion-battery (KIB) systems currently [8,9]. Compared to sodium, potassium also has a substantial abundance and even lower reduction potential (-2.94 V for K^+/K vs. -2.71 V for Na^+/Na) [8]. Some significant progress has been made on KIBs, such as cathode materials, anode materials and liquid-organic electrolytes [10–12].

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Like LIBs or NIBs, developing electrode materials is a critical step for exploiting high-performance KIBs [13,14]. Organic electrode materials provide a new avenue to develop high-capacity energy storage systems and have been thoroughly studied in Li-ion batteries [15–19]. Furthermore, the structure diversity, low cost, good recyclability and environmental friendliness of organic materials have led to heightened battery-related research efforts [20–23]. Among these organic electrode materials, PTCDA has been studied as a cathode material for lithium-ion batteries, sodium-ion batteries and potassium-ion batteries [9,24,25]. In Chen's work, they demonstrated a PTCDA cathode with excellent electrochemical performance for PIBs [8]. However, the dissolution of the organic active materials in liquid electrolytes, irreversible reaction and low conductivity currently results in critical safety and poor cyclicality problems [26–28]. Moreover, the employ of flammable liquid electrolytes in K-ion batteries draws significant safety concerns as well as in the case of LIBs and NIBs.

In an attempt to solve the discussed issue, the use of solid state batteries is an effective way [27,29–32]. PPC is one of the new-type polycarbonates that have attracted substantial interests due to its environmental friendliness [33–35]. Cellulose is also known to be a renewable material and displays marvelous properties such as chemical stability and thermal stability. In LIBs, Cui's group presented a brilliant strategy to use PPC as ionic transit material and develop cellulose nonwoven membrane as the backbone [34]. The LiFePO₄/Li cells exhibit a high capacity of 142 mA h g⁻¹ at 0.1C at room temperature. Inspired by these works, we herein report on a room-temperature PPC-based solid polymer electrolyte with cellulose nonwoven backbone (PPCB-SPE) designed for a high safety solid-state K-ion battery with an organic cathode for the first time. The PPCB-SPE delivers a high conductivity of 1.36 × 10⁻⁵ S cm⁻¹ at 20 °C. Furthermore, the novel all-solid-state KIBs displays a high capacity of 118 mA h g⁻¹ and good cycling performance at room temperature.

2. Experimental

2.1. Chemical and materials

PPC utilized in this work was purchased from Sigma-Aldrich. Battery-grade KFSI was supplied by Tokyo Chemical Industry Co. Ltd. Tetrahydrofuran was commercially available. The cathode material PTCDA was purchased from J&K. The chemistry analytical filter paper was used as cellulose nonwoven backbone, bought from Hangzhou Special Paper Co. Ltd. The organic-liquid electrolyte for PIBs is self-made with a component of 1M KFSI in EC/DEC (v/v, 1:1).

2.2. Preparation of PPC-based solid polymer electrolyte

PPCB-SPE was synthesized by a facile solution-casting method reported previously [34]. Predetermined amounts of PPC and KFSI were dissolved in tetrahydrofuran to obtain a homogenous solution. The concentration of PPC in tetrahydrofuran is 15 wt%. In addition, the concentration of K salt varies from 12 to 36 wt% (x wt%: x = (m[KFSI]/m[PPC] × 100). Subsequently, the pre-dried cellulose nonwoven membranes (16.5 mm in diameter) were immersed in the as-prepared solution for 30 min. Following, cellulose nonwoven membranes were removed to a Teflon plate and were dried in a vacuum oven at 60 °C for 24 h. Finally, the PPCB-SPE membranes were stored in an argon-filled glove box.

2.3. Characterization methods

The micro-structure of the commercial cathode material (PTCDA) was examined via X-ray diffractometer (XRD) on a Rigaku Dmaxc diffractometer by using Cu K α radiation at a scanning rate of 10 °C/min from 10 °C to 40 °C. SU-70 field emission scanning electron microscopy (FESEM) was employed to evaluate the micro-morphologies of PTCDA

and cellulose nonwoven membrane with or without PPC-18 wt% KFSI.

2.4. Electrochemical measurements

The ionic conductivities of PPCB-SPE-x wt% KFSI at room temperature and PPCB-SPE-18 wt% KFSI at 20–120 °C was measured by EIS at a frequency range of 1–10⁶ Hz using a CHI660E electrochemical workstation. The PPCB-SPE membranes were sandwiched between two stainless-steel electrodes in 2016 coin cells; the cells for EIS test were kept at each temperature for 25 min to reach thermal balance. The bulk resistance of the PPCB-SPE membranes were confirmed from the impedance spectrum. The ionic conductivities were counted from Equation (1)

$$\sigma = L/R_b S \quad (1)$$

where S and L are the area and thickness of the SPE membranes respectively and R_b is the bulk resistance. The electrochemical window of PPCB-SPE was tested by using an electrochemical workstation in the potential range from 1 V to 5 V at a scanning rate of 0.5 mV s⁻¹ with potassium metal foil as a reference and counter electrode and a stainless steel as working electrode. The cyclic voltammetry of K plating/stripping on stainless steel was also tested using the CHI660E electrochemical workstation at room temperature.

The cathode electrode consists of 60 wt% PTCDA, 20 wt% PVDF and 20 wt% carbon black (Super-P). The mixture was milled in N-methyl pyrrolidone (NMP) solvent for 16 h. The resultant viscous slurry was casted on aluminum current collector and dried at 90 °C for 24 h in a vacuum oven. Following, the electrode films were punched into discs of 14 mm in diameter. The loading of the cathode is about 1.2–1.4 mg/cm². The CR2016 coin K cells were assembled by using a K metal foil, a PTCDA cathode and a K⁺-conducting PPCB-SPE without a separator (or organic-liquid electrolyte). The cell fabrication was performed in an argon-filled glove box (H₂O < 0.1 ppm, O₂ < 0.1 ppm). The electrochemical performance was tested and found to be between 1.5 and 3.5 V at different current densities by a computer-controlled programmable battery charger.

3. Results and discussions

The scanning electron microscopy (SEM) images of the as-prepared cellulose nonwoven membrane and PPCB-SPE provide insight into their morphology. As revealed in Fig. 1a, the cellulose nonwoven membrane comprises randomly distributing stout fibers. The average diameter of these fibers is about 20 μ m. These fibers formed a continuous and tenacious framework by twinning and interlocking with each other, thus they can support the solid electrolyte with sufficient mechanical strength. Furthermore, there are interconnecting pores inside the fibre grid to carry an adequate amount of solid electrolyte. Fig. 1b shows the surface morphology of PPCB-SPE. After compounding solid electrolyte into the cellulose framework, a glossy and homogeneous surface of PPCB-SPE was obtained and the pores inside the framework were filled up with solid electrolyte. As shown in the cross-section image (Fig. S1), the composite has a continuous structure. Therefore, by compounding PPCB-SPE into the framework of cellulose nonwoven membrane, a three-dimensional continuous and homogeneous all-solid-state electrolyte was obtained. The as-prepared solid electrolyte with this structure could possibly eliminate the safety issues of the batteries with organic electrolyte.

The behavior of potassium metal stripping/plating on stainless steel across the solid polymer electrolyte was tested via cyclic voltammetry with K metal as both the reference and counter electrode and stainless steel as the working electrode. Fig. 1c shows a pair of strong reduction and oxidation peaks. The sharp reduction peak in the negative scan at -0.3 V (vs. K/K⁺) reveals the plating of potassium on working electrode. In the subsequent positive scan, the oxidation peak at 0.21 V (vs.

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