



Flexible thin-film battery based on solid-like ionic liquid-polymer electrolyte



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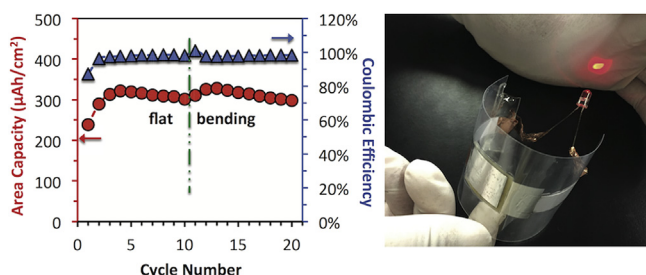
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HIGHLIGHTS

- Solid-like gel polymer electrolyte was fabricated containing 60 wt% ionic liquid.
- The incorporation of nonvolatile ionic liquid offers enhanced safety and stability.
- A low-cost, scalable lamination method was employed to fabricate flexible LIBs.
- The flexible LIB could deliver a stable capacity in flat and bent configurations.

GRAPHICAL ABSTRACT



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ABSTRACT

The development of high-performance flexible batteries is imperative for several contemporary applications including flexible electronics, wearable sensors and implantable medical devices. However, traditional organic liquid-based electrolytes are not ideal for flexible batteries due to their inherent safety and stability issues. In this study, a non-volatile, non-flammable and safe ionic liquid (IL)-based polymer electrolyte film with solid-like feature is fabricated and incorporated in a flexible lithium ion battery. The ionic liquid is 1-Ethyl-3-methylimidazolium dicyanamide (EMIMDCA) and the polymer is composed of poly(vinylidene fluoride-co-hexafluoropropene) (PVDF-HFP). The electrolyte exhibits good thermal stability (i.e. no weight loss up to 300 °C) and relatively high ionic conductivity ($6 \times 10^{-4} \text{ S cm}^{-1}$). The flexible thin-film lithium ion battery based on solid-like electrolyte film is encapsulated using a thermal-lamination process and demonstrates excellent electrochemical performance, in both flat and bent configurations.

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1. Introduction

Flexible energy storage devices offer many advantages including compatibility with the flexible electronics and flexible applications [1]; for example, they can be attached to the biological organs or

embedded in textiles/clothing. Among the various energy storage devices, lithium-ion batteries (LIBs) are ideal candidates to transform into flexible devices, due to their superior attributes including higher energy density and efficiency. Over the past several decades several structural and materials designs for the fabrication of flexible LIBs have been proposed [2–10]. Min Koo et al. developed an all-solid-state bendable lithium-ion battery based on ceramic electrolyte exhibiting a capacity of $106 \mu\text{Ah/cm}^2$ [2]. Liangbing Hu et al. designed a paper-based lithium ion battery which employed

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carbon nanotubes instead of the traditional metals as the current collector [3]. Sheng Xu and others proposed self-similar interconnects to pack a set of cells forming a highly stretchable battery [4]. Comprehensive surveys of this topic can be found in two recent reviews by Lee et al. [11] and Zhou et al. [12].

In designing flexible LIBs, electrolytes play a crucial role. The common liquid electrolytes – used in many pouch-type flexible batteries – are less than ideal due to the risk of leakage and lack of mechanical stability. On the other hand, solid electrolytes are superior in terms of stability and safety but their relatively low ionic conductivity results in insufficient electrochemical performance and power density. In order to develop high-performance LIBs with relatively safer electrolyte, compared to organic liquids, intense efforts have been directed toward the development of gel electrolytes. Gel polymer electrolytes are usually obtained by infusing a large amount of liquid electrolyte into a suitable polymer matrix [13,14]. The role of the polymer matrix is mainly to store the liquid electrolyte and provide mechanical support. The ionic conductivity of a gel electrolyte, governed mainly by the conductivity and amount of the liquid phase, is relatively high and can be competitive with respect to the traditional organic liquid electrolytes. Various polymers including poly(ethylene oxide) (PEO) [15,16], polyacrylonitrile (PAN) [17,18], poly(methyl methacrylate) (PMMA) [19,20], and poly(vinylidene fluoride-co-hexafluoropropene) (PVDF-HFP) [21,22], have been studied as potential matrices for gel electrolyte. Among these polymers, PVDF-HFP-based gel electrolytes exhibit excellent mechanical properties as well as high ionic conductivity ($\sim 10^{-3}$ S cm^{-1}) and have become the leading polymer matrix materials in this field.

Unfortunately, because of the presence of reactive, volatile, and flammable organic solvents (e.g. ethylene carbonate (EC) and propylene carbonate (PC)), common gel electrolytes share some of the same safety concerns as associated with liquid electrolytes [23]. To further enhance the safety of the gel electrolytes, ionic liquids can be introduced in the polymer matrix to replace the organic liquid phase [24,25]. Ionic liquids are essentially low-melting temperature salts that remain in liquid phase at room temperature. They possess several favorable properties such as high ionic conductivity, non-volatility, non-flammability, and thermal, chemical stability [26]. Many attempts have been made to replace the traditional organic carbonates with ionic liquids in the solvent-salt and gel electrolytes [27–33]. However, as discussed in a critical review, batteries with these ionic liquid-based liquid or gel electrolytes usually exhibit poor cyclic and power performance compared to those made with conventional organic solvent-based electrolytes even though they exhibit similar ionic conductivities [34].

In order to achieve high ionic conductivity, both the cation and anion of the ionic liquid should be generally small and carry a well-delocalized charge [35]. Among many ionic liquids, 1-Ethyl-3-methylimidazolium dicyanamide (EMIMDCA) has a relatively high ionic conductivity (27 mS/cm), however, the electrochemical window is relatively small (2.9 V). The high ion conductivity can be attributed to the small size of the dicyanamide anion [36,37]. Adding lithium salt can slightly increase the viscosity and thus decrease the ionic conductivity of ionic liquid [38].

In this study, EMIMDCA ionic liquid based polymer electrolyte film with solid-like feature is fabricated and incorporated into a flexible Li ion battery. The flexible battery demonstrates excellent electrochemical performance, in both flat and bent positions.

2. Experimental

2.1. Preparation of the gel electrolyte

The weight ratios of the gel electrolyte components consist of

30% PVDF-HFP (polymer matrix), 60% EMIMDCA (ionic liquid), and 10% LiClO₄ (salt). LiClO₄ salt has been used mainly because of its stability, better tolerance to moisture, and lower cost. The gel electrolyte was prepared by the general solution-casting method: 1) 0.6 g PVDF-HFP (Mw = 400,000, purchased from Sigma Aldrich) was added in 20 mL N,N-Dimethylformamide (purity 99.8%, Sigma Aldrich) and the mixture was stirred at about 80 °C for 1 h; the weights of other materials were calculated based on the weight of PVDF-HFP; 2) after the PVDF-HFP was dissolved, the EMIMDCA (Sigma Aldrich) and LiClO₄ (Sigma Aldrich) were added to the solution and stirred at 50 °C overnight (about 12 h); 3) the viscous solution was poured into a glass petri-dish and placed in the vacuum oven at 50 °C for 24 h to allow the solvent to evaporate. The obtained thin-film electrolyte with an averaged thickness of about 0.2 mm was stored in the dry glove box to prevent moisture absorption.

2.2. Characterizations of the electrolytes

The electrochemical impedance spectroscopy of the PVDF-IL electrolyte was obtained using Metrohm Autolab with frequency response analysis (FRA) module. The sample was sandwiched between two stainless steel electrode discs and the complex impedance spectra were obtained in the frequency range of 10 Hz–500 kHz. Thermogravimetric analysis (TGA) results were collected using Q50 TGA (TA Instruments) in the temperature region of 20 °C–600 °C. The temperature scanning rate was 10 °C min^{-1} .

2.3. Battery assembly and testing

The anode was prepared by coating the slurry (Li₄Ti₅O₁₂: Carbon black: PVDF = 8:1:1 dissolved in N-Methyl-2-pyrrolidone; purchased from MTI Corporation) on the aluminum foil and dried in the vacuum oven at 80 °C for 24 h. Since the Li₄Ti₅O₁₂ anode has a higher potential for Li-intercalation (~ 1.5 v.s. Li⁺/Li), it can be used with an aluminum foil current collector. The cathode (LiCoO₂ on aluminum foil with mass loading of about 12 mg/cm²) was purchased from MTI Corporation. Both the cathode and anode were cut into 1.5 cm × 1.5 cm squares and a copper strip was placed on the aluminum side of each electrode for electrical connection to the external devices. A piece of the PVDF-IL electrolyte was sandwiched between the cathode and anode and one drop (about 2 mg) of 1M LiClO₄ in EMIMDCA was poured on the surface of each electrode to further reduce the interfacial resistance. This step is critical in reducing the interfacial resistance in the battery. Then, the battery was placed on a thermal-adhesive plastic laminating sheet and the laminating machine (Saturn SL-95 Laminating Machine, Amazon) was used to laminate the battery. After the lamination process, the extra parts of the sheet were cut and removed, and the copper strips were exposed for battery testing. The cyclic voltammograms of the battery were obtained using Metrohm Autolab at a scan rate of 0.05 mV/s from 1.5 V to 2.8 V. The cycle performance of the battery was tested by Arbin battery test equipment at a constant current density of 35 mA/mg (0.25 C of LiCoO₂) in the voltage range of 1.6 V–2.7 V. To evaluate the electrochemical performance of the laminated battery in bending condition, the battery was wrapped around a 2 cm radius cylinder (Fig. S4).

3. Results and discussion

Fig. 1a shows a freestanding gel electrolyte film, with solid-like appearance, composed of polyvinylidene fluoride-co-hexafluoropropene (PVDF-HFP), 1-Ethyl-3-methylimidazolium dicyanamide (EMIMDCA) ionic liquid (Fig. S1), and LiClO₄ lithium

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