Electrospun Polyacrylonitrile Microfiber Separators for Ionic Liquid Electrolytes in Li-ion Batteries

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Abstract

Despite much recent progress in the development of room temperature ionic liquid (RTIL) electrolytes for lithium-ion batteries (LIBs), relatively little work has been done in terms of investigating commercially applicable separator materials capable of accommodating RTILs. In this work, we demonstrate an electrospun polyacrylonitrile (PAN) microfiber separator. The PAN microfiber separators show high degrees of porosity (~83%), wettability, and mechanical strength ($\sigma_{UTS} = 16.98$ MPa and $E = 5.95$ MPa). The physical properties of our electrospun separators lead to impressive electrochemical performance, showing an **apparent** MacMullin number (N_M) of <5 when combined with the $\text{PYR}_{13}\text{FSI}$ (1.2M LiFSI) electrolyte. These results are validated by superior rate performance and the exhibition of a high capacity full-cell utilizing a PAN microfiber separator in combination with the $\text{PYR}_{13}\text{FSI}$ (1.2M LiFSI) RTIL electrolyte. Such work represents significant progress in the advancement of RTIL electrolytes for LIBs, indicating that nonwoven separators are a commercially viable solution to the previous lack of separator materials for RTIL electrolytes.

Keywords: (ionic liquids, electrolyte, polyacrylonitrile, battery separator, electrospinning)

1. Introduction

While commercialized lithium-ion batteries (LIBs) are capable of achieving energy-densities of up to 800 Wh L^{-1} in the 18650-type cell configuration, the advent of electrified transportation and renewable energy technologies demands safer and higher performance energy storage. In the search for the next generation LIB, researchers are developing new materials, modifying existing materials, and investigating unique combinations of materials. One such approach towards a safer, higher energy-density battery involves replacing the conventional

carbonate-based electrolyte with a room temperature ionic liquid (RTIL) electrolyte system. RTIL materials have attracted attention in electrochemical applications because of their non-flammability, negligible vapor pressures, high thermal and electrochemical stabilities, and adequate ionic conductivities.[1-3] Utilizing a RTIL electrolyte induces new surface and reaction chemistries, recently showing success in demonstrating high performance Li-ion cells.

RTIL electrolytes based on the bis(fluorosulfonyl)imide (FSI) or bis(trifluoromethanesulfonyl)imide (TFSI) anion show particularly advantageous properties in Li-ion cells.[4-8] For example, TFSI has been shown to mitigate sulfur dissolution and polysulfide redox shuttle in sulfur-based electrodes,[9,10] FSI shows strong compatibility with $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ cathodes [11] and graphite anodes,[5,12] and both anions allow good reversibility of the Li metal electrode.[13,14] While much progress has been made in regards to developing high energy-density electrode-RTIL systems, research has overlooked one major hurdle for the commercialization of RTILs as LIB electrolytes: the separator material. Most reports on RTIL electrolytes for LIBs use thick (*ca.* 300 μm) glass fiber separators that are too brittle for incorporation in the conventional jelly-roll configuration.

In short, commercial separator materials are incompatible with RTIL electrolytes. Commercially produced batteries utilize thin polyolefin separators, such as Celgard®'s polypropylene (PP) membranes. Such separators have small pore sizes ($< 1 \mu\text{m}$ pore diameter); due to their high viscosities, RTIL electrolytes are unable to sufficiently wet these separators. In order to develop an RTIL electrolyte membrane that is compatible with both commercially attractive electrode materials and the winding processes in the existing LIB manufacturing infrastructure, a new separator material should be developed. One recent study by M. Kirchhöfer et al. studied different RTIL electrolytes in combination with various separator materials and

their compatibilities with the Li metal electrode.[15] While this study provides insight into alternative separator materials specifically for RTIL electrolytes, a separator must be developed that can accommodate the electrochemistry of RTILs while maintaining adequate mechanical properties for employment in the jelly-roll cell configuration (ie; 18650-type cells).

To this end, we turn to electrospun polyacrylonitrile (PAN) microfiber mats. PAN based separators have been studied as alternatives to conventional microporous polymer materials,[16-23] but all previous work related to PAN nonwoven separators was tailored to the utilization of conventional organic electrolyte. A separator material must be electronically insulating, allow for minimal ionic resistance, possess high mechanical and dimensional stability, have sufficient physical strength to allow for easy handling, possess chemical resistance to degradation by electrolyte impurities, allow for easy wetting by the electrolyte, and be inert against the electrode materials.[17] Based on previous demonstration of nonwoven separators' superior ionic transport properties and the ability to tune the separators' physical properties via the electrospinning fabrication process, we identified PAN fiber mats as a candidate separator material for RTIL electrolytes.

In this study, we have developed an electrospun PAN microfiber separator that is highly compatible with RTIL electrolytes and well-suited to the winding processes in commercial battery manufacturing. We report the major physical, mechanical, and electrochemical properties of the separator and ultimately demonstrate the separator in a high capacity Li-ion full-cell.

2. Experimental

2.1 Fabrication of PAN microfiber separators

PAN microfiber-based nonwoven separators were prepared via electrospinning of a PAN/N,N-dimethylformamide (DMF) solution. A 10 wt. % solution was prepared by dissolving

polyacrylonitrile (PAN, Sigma Aldrich) in DMF. This solution was ejected through a voltage applied (27 kV) nozzle to the grounded target plate with a feeding rate of 1.5 mL h⁻¹. The distance between the nozzle and the grounded target was 32 cm. Electrospinning for 2 h resulted in circular fiber mats with a thickness of approximately 200 μ m and a diameter of approximately 13 μ m.

2.2 Physical/ mechanical characterization of PAN microfiber separators

Morphological analysis of the as-made and post-test (100 charge-discharge cycles in a Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ half-cell) electrospun PAN fiber separators was carried out using low-vacuum scanning electron microscopy (JEOL SEM 6840LV). The wettabilities of the PAN separators, a conventional polyolefin microporous separator (PP, Celgard), and a glass fiber separator (GF/F, Whatman) by both ionic liquid electrolytes and organic electrolyte were estimated by monitoring the variation of contact angle between the liquid electrolyte and the membranes at every 0.01 s using a high shutter speed camera. Porosity of the PAN fiber separators was determined using the Archimedes Technique, also known as the Immersion Technique. PAN microfiber mats were punched into the planar dog-bone configuration (20 mm length) for tensile testing (Shimadzu MMT-500 N, Japan) at a strain rate of 2.5 x 10⁻⁴ s⁻¹, with a 100% strain corresponding to 20 mm.

2.3 Electrochemical characterization PAN microfiber separators

RTIL electrolytes consisted of the N-methyl-N-propyl-pyrrolidinium (PYR₁₃⁺) cation and either the FSI⁻ or TFSI⁻ anion. The RTIL electrolytes studied were PYR₁₃FSI (1.2M LiFSI), PYR₁₃TFSI (0.6M LiTFSI), and 1M LiPF₆ in ethylene carbonate, diethyl carbonate (50:50, Soulbrain) was used as a conventional organic electrolyte. Ionic liquid solutions were provided

by Boulder Ionics Corporation (U.S.A.) and scanned for halide impurities. Impurities (F^- , Cl^- , Br^- , SO_4^-) were quantified using a Dionex ICS-1100 ion chromatograph, calibrated for sensitivities as low as 1 ppm. All ionic liquids and lithium salts used in this study were subjected to ion chromatography, and the total impurity content of every solution prepared was calculated based off the mass percentage of electrolyte component in the total mass of the electrolyte. The solutions contained less than 20 ppm (w/w) of moisture and less than 10 ppm (w/w) of halide and metal-ion impurities. σ_{ionic} was determined for the electrolytes using the standard complex impedance method on a Solartron 1280C for a frequency range of 20 kHz - 10 mHz.

A symmetric rate study (charge rate=discharge rate) was carried out using $Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ positive electrodes and lithium foil negative electrodes with the cycling protocol outlined in a previous study, in which three cycles were performed at each rate (C/10, C/5, 1C, 2C, 4C, and 8C) to study cycling stability at higher rates and capacity recovery upon lowering rate.[11] Composite electrodes were fabricated using slurry coating of $Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ powder material (Johnson Controls), acetylene black (Alfa Aesar, AB) as a conductive additive, and polyvinylidene fluoride (Kynar, PVDF) binder in a weight ratio of (85:7.5:7.5). The composite was cast onto clean, high-grade aluminum foil current collector using a 5 mil doctor blade to achieve an active material mass loading of approximately 3 mg cm^{-2} . The electrodes were dried for 12 h at 80 °C. In order to ensure adequate electrical contact and to improve overall energy density, the electrodes were calendared to 75% of their initial thickness. Electrode disks of 1.27 cm diameter were punched and then dried at 120 °C in a vacuum oven for 12 hours before testing. PAN separators were soaked in RTIL electrolyte for 12 hours prior to fabrication of full-cells. Full-cells were built and tested according to the procedure outlined in our previous work, in which each electrode were electrochemically pre-conditioned

prior to placing in the full-cell to allow precise control of the amount of lithium in the system.[24] Calculated from the active material mass, nSi-cPAN anodes were fabricated and matched with L333 cathodes such that the total anode capacity was 160% of that of the cathode capacity. Both electrodes were then pre-conditioned: the anodes were allowed to run for 10 charge-discharge cycles in a half-cell configuration and were stopped after full lithiation, while the cathodes were allowed to run for 3 charge-discharge cycles in a half-cell configuration and were stopped after full delithiation. The half-cells were then disassembled and the electrodes were used to fabricate 2032 coin-cell (Al-clad cathode cup) type full-cells. This method of pre-conditioning allows for full control of the amount of lithium in the system.

3. Results and discussion

3.1 PAN microfiber separator morphology, electrolyte compatibility, and mechanical properties

Building on the previous studies of PAN nonwoven separators for utilization in electrochemical cells with conventional organic electrolytes, this work proves that PAN separators are a strong candidate for RTIL based membranes. While the literature suggests a fiber diameter of approximately 500 nm or less, as controlled by electrospinning solution flow rate and electric field strength,[25] allows for optimal performance with conventional organic electrolyte materials, it is proposed that a different microstructure is necessary to accommodate an RTIL electrolyte. For this reason, we aimed to develop a separator with a larger average pore size in order to allow for high wettability with a more viscous electrolyte solution.

Figure 1 shows a high-resolution scanning electron microscope (SEM) image of our PAN fiber mat as made, showing a relatively uniform fiber diameter of approximately 1 μm . The

morphology of this mat is significantly different than those previously demonstrated, with much thicker fiber diameters. It was proposed that the larger fiber diameter, which leads to higher porosity, should allow for adequate wettability by a RTIL electrolyte.

The porosity of the nonwoven PAN separator designed in this work was determined both experimentally and theoretically. The porosity of a wide variety of materials is determined by the Archimedes Technique, also known as the Immersion Technique, using Equation 1:

$$P_{exp} = \frac{m_{sat} - m_{dry}}{m_{sat} - m_{sus}} \quad (1)$$

where m_{dry} is the mass of the dry material, m_{sat} is the mass of the material after becoming saturated with the liquid, and m_{sus} is the mass of the material when suspended in the liquid. De-ionized water was chosen as a suitable immersion medium for the PAN separators. Using this method, the porosity of the nonwoven PAN separator mat was determined to be 83%. The porosity of the PAN separator was also calculated using the known density of electrospun PAN (1.12 g cm^{-3}) and the mass and volume of a punched and dried PAN separator sheet ($P_{theoretical} = V_{PAN}/V_{separator}$). Using this method, the porosity of the nonwoven PAN separator was determined to be 84.66%, thereby substantiating the experimentally determined value. This is much higher than the Celgard PP separator porosity of 39% and meets the known requirement for battery separator porosity of at least 35%. This data also conforms to the trend found in previously studied nonwoven separators in which larger fiber diameters result in a higher degree of porosity.[19] PAN separators with fiber diameters of <500 nm have been shown to possess porosities of up to 76%.[19] This data suggests that the PAN separators fabricated in this work may be better suited to wetting with an RTIL electrolyte.

The wettability of our PAN separator was estimated using high-speed photography and compared to the wettability of a PP separator and glass fiber separator by two RTIL electrolytes and a conventional organic electrolyte. Electrolytes used include $\text{PYR}_{13}\text{FSI}$ (1.2M LiFSI), the more viscous RTIL $\text{PYR}_{13}\text{TFSI}$ (0.6M LiTFSI), and the conventional organic electrolyte EC/DEC (1M LiPF_6). The results of our wettability study are shown in Figure 2. A lower contact angle is indicative of a higher degree of wettability, and a faster change in contact angle suggests a higher rate of wetting. In all cases, the nonwoven PAN separators showed the highest wettability, with the Celgard PP membrane proving to be the least wettable. Good wettability proves the separator's ability to effectively wick electrolyte, thereby facilitating smooth diffusion of the electrolyte during assembly of an electrochemical cell. Thus, these results further suggest that the PAN nonwoven separators are a strong candidate for utilization with an RTIL electrolyte.

In addition to the physical properties of the electrospun PAN nonwoven separators, it is important to study their mechanical properties. The separators used in spiral wound cells (conventional design) require high tensile strength because the separator is wound together with the electrodes under tension; the separator must not elongate significantly under tension in order to avoid contraction in the direction perpendicular to winding.[17] Commercial separators require a tensile strength corresponding to 2% stretch (strain) at 1000 psi, or 6.89 MPa.[17] To this end, the tensile strength of our PAN separator mats was determined and compared to that of Celgard PP and Whatman GF/F separators. The results of the tensile tests are reported in Figure 3. The tensile strength of the PP separator coincides well with previous academic reports of the tensile strength of similar materials.[26] While the PAN separator's ultimate tensile strength (σ_{UTS}) and yield strength (σ_{yield}) are lower than that of the PP separator, they are much higher

than the σ_{UTS} and σ_{yield} of the glass fiber separator and meet the aforementioned requirements for a commercial separator. Our PAN microfiber separators show a σ_{UTS} of approximately 16.39 MPa. Moreover, the PAN separators showed the highest elastic modulus (E), with $E = 5.95$ MPa, of the separator materials tested, suggesting a lower degree of stretch in the elastic region for a given stress. This compares to $E = 4.01$ MPa for the PP separator, and $E = 0.319$ for the glass fiber separator. The combination of physical and mechanical characterization demonstrated in this study substantiates our claim that electrospun PAN fiber separators are a feasible choice for employment in Li-ion cells containing RTIL electrolyte.

3.2 Electrochemical performance of Li-ion cells using PAN microfiber separators

Nonwoven separators have exhibited excellent battery performance with carbonate-based electrolytes in terms of cycling life and high rate capability. Because of their high degrees of porosity and wettabilities, nonwoven separators provide a less tortuous path for Li^+ transport and therefore show lower ionic resistances than polyolefin separator materials.[19] In order to demonstrate the adequate ionic conductivity of the RTIL-PAN separator system developed in this work, the system's **apparent** MacMullin number (N_M) was calculated and compared to that of a conventional system. **The** N_M is defined as the ratio of the conductivity of the pure electrolyte (σ_0) to that of the **immersed** separator (σ_{eff}) as shown in Equation 2:[19]

$$N_M = \frac{\sigma_0}{\sigma_{\text{eff}}} \quad (2)$$

A lower **apparent** N_M infers higher rate and power capability. Commercial separator systems possess **apparent** N_M values of ≤ 12 . [17,19] The PAN microfiber separator **immersed in** RTIL electrolyte shows a relatively low N_M of 4.96, well within the bounds of the commercial standard. It is therefore expected that our electrospun PAN separators should allow for high rate performance in Li-ion cells.

A rate study of a $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ half-cell was carried out in order to examine the rate performance using a PAN separator with RTIL electrolyte, and the results of this experiment were compared to the same rate study using a Whatman GF/F separator. The rate study and cycling stability results are presented in Figure 4. The nonwoven PAN separator allows for high capacities at all rates tested as well as high cycling stability. These results corroborate similar studies of nonwoven separators used in combination with organic electrolytes in which the better cycling performances shown when using nonwoven separators are attributed to their higher porosities and the subsequent enhancements in ionic conductivity and uptake of liquid electrolyte [18,19]; **as shown in Figure 1, the PAN microfiber mat possesses a very open microstructure that facilitates better uptake of electrolyte.** The high cycling stability suggests that the PAN microfiber mat is electrochemically stable against the electrode materials utilized and the electrolyte, as side reactions that might occur within the cycling voltage range would induce capacity instabilities. A post-cycling morphological analysis was conducted on a separate but identical half-cell allowed to cycle 100 times at a rate of 1C. SEM imaging of the PAN separator after cycling, presented in Figure 5, **reveals physical change in the separator after cycling.** It is proposed that the tighter packing of the fibers, as compared to the as-made PAN microfiber mat, is due to compaction during fabrication and cycling of the cell in a coin-type configuration.

Swelling due to absorption of electrolyte may have also occurred. It is noted that such changes have no detrimental effects on performance as proven in the cycling experiments.

Finally, we demonstrate our electrospun PAN microfiber separator in a high-capacity nSi-cPAN/Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ full-cell developed in a recent report.[9] The cycling stability observed in this experiment validates our claim that a nonwoven separator is an adequate replacement for conventional polyolefin materials in RTIL-based LIBs. Demonstration of the PAN microfiber separator in a full-cell is the ultimate test of its electrochemical and physical stability, proving the chemical stability of the separator and its compatibility with commercially attractive electrode materials. While a full safety and thermal stability analysis should be completed for the RTIL-PAN separator system, this combination of data provides strong evidence for the compatibility of PAN microfiber separators with a RTIL-based battery. Thus, this work represents important progress in the field of RTIL electrolytes and demonstrates their potential for commercialization.

4. Conclusions

In this paper, we have developed a separator material compatible with both RTIL electrolytes and the commercial LIB manufacturing infrastructure for 18650 cells. Based on the established commercial electrospinning process, PAN microfiber separators present a viable alternative to polyolefin materials. Most importantly, electrospun PAN microfiber separators show superior physical, mechanical, and electrochemical properties when used in combination with RTIL electrolytes.

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Figure Captions

Figure 1. SEM micrograph of the as-made electrospun PAN microfiber mat.

Figure 2. Wettabilities of PP, glass fiber, and electrospun PAN microfiber separators by EC/DEC (1M LiPF₆) (a), PYR₁₃FSI (1.2M LiFSI) (b), and PYR₁₃TFSI (0.6M LiTFSI) (c), as estimated by measuring change in electrolyte droplet-separator plane contact angle over time.

Figure 3. Tensile tests of PP, glass fiber, and electrospun PAN microfiber separators. Inset shows detail for the glass fiber and PAN microfiber separator tests.

Figure 4. Rate study of Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O₂ half-cells containing PYR₁₃FSI (1.2M LiFSI) comparing performance of a glass fiber separator and the electrospun PAN microfiber separator. Electrochemical cycling was performed at room temperature between 3-4.2V vs. Li/Li⁺.

Figure 5. SEM micrograph of the electrospun PAN microfiber separator after 100 cycles in a $\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ half-cell.

Figure 6. Cycling of a $\text{nSi-cPAN/Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2$ full-cell utilizing $\text{PYR}_{13}\text{FSI}$ ($1.2M$ LiFSI) electrolyte and the electrospun PAN microfiber separator. Electrochemical cycling was performed at room temperature between 2-4.15 V. Specific capacities are normalized to total active material weight ($\text{Li}(\text{Ni}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3})\text{O}_2 + \text{Si}$).

Graphical Abstract