

Electronic Supplementary Information

“Rapid, Microwave-Assisted Thermal Polymerization of Poly(Ethylene Glycol) Diacrylate-Supported Ionogels”

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Experimental Procedure

1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide (EMI TFSI) was purchased from EMD Chemicals, Inc. Poly(ethylene glycol) diacrylate (PEGDA, average M_n 575 g mol⁻¹), and 2,2'-azobis(2-methylpropionitrile) (AIBN) were purchased from Sigma-Aldrich. All chemicals were used as received.

An ionogel precursor solution containing the ionic liquid (EMI TFSI), PEGDA, and AIBN was prepared by weighing components on a balance. The precursor solution PEGDA composition was 25 wt%; the solution contained approximately 0.5 wt% AIBN, with the balance made up by EMI TFSI. The solution was sonicated for 30 minutes to degas and mix (Branson 2510, 2.8L). A small PTFE (Teflon) block with a rectangular cavity (1.177 cm length, 0.285 cm width, 1.000 cm depth) was employed as a mold. 100 μ L of precursor solution was deposited into the mold, filling approximately one-third of the cavity volume. The mold/precursor solution assembly was placed into a General Electric domestic 1150 W microwave oven (model JES1451) located inside a standard chemical fume hood. Power level 2 (described by the operating manual to provide approximately 20% of the maximum microwave power) was employed to heat the assembly using “on” pulses of 5 s (each followed by an “off” period of $\sim$ 5 s) for a total of 10 s to 25 s. Higher power level settings or longer “on” pulses resulted in thermal decomposition of the solution (indicated by a dark discoloration and/or burning odor). By recording the masses of the precursor solution (before heating) and the resulting ionogel, several replicate trials revealed an average mass loss of less than 1%. This indicates that there is no significant volatilization of the PEGDA monomer during the microwave heating process.

Characterization Details

FTIR spectroscopy was employed to investigate the extent of PEGDA polymerization/cross-linking. Spectra were collected using a JASCO Corporation FT-IR-6200 by conducting 50 scans with a 2 cm⁻¹ resolution.

AC impedance spectroscopy measurements were recorded using a Princeton Applied Research VersaSTAT 3 potentiostat with a built-in frequency response analyzer. A two-electrode set-up employed, with the electrolyte located between glassy carbon electrodes. Two-electrode device

impedance was measured over the frequency range 1 Hz to 1 MHz with a sinusoidal voltage amplitude of 10 mV, superimposed on 0 V DC vs. open circuit. Two-electrode cyclic voltammetry was performed using the same VersaSTAT 3 potentiostat at two voltage sweep rates (0.1 V s^{-1} and 1.0 V s^{-1}) within a window of -1.5 V to 1.5 V vs. open circuit. All electrical characterization was performed at room temperature ($22 \text{ }^{\circ}\text{C}$) under ambient laboratory conditions.

Mechanical response data were acquired by unconfined compression testing using a dynamic mechanical analyzer (RSA3, TA Instruments) in free extension mode. Compressive loading and unloading tests were performed on a manually defined portion of the ionogel following electrical characterization, in order to comply with the sample thickness constraint of the instrument (no more than 1.5 mm). The rate of strain applied was 0.1% per second to an upper limit of approximately 15% strain. A Young's modulus value was calculated from a best-fit line to the resulting stress-strain data within the 5%–10% strain region. It was assumed that the cross-sectional area did not change significantly during testing.

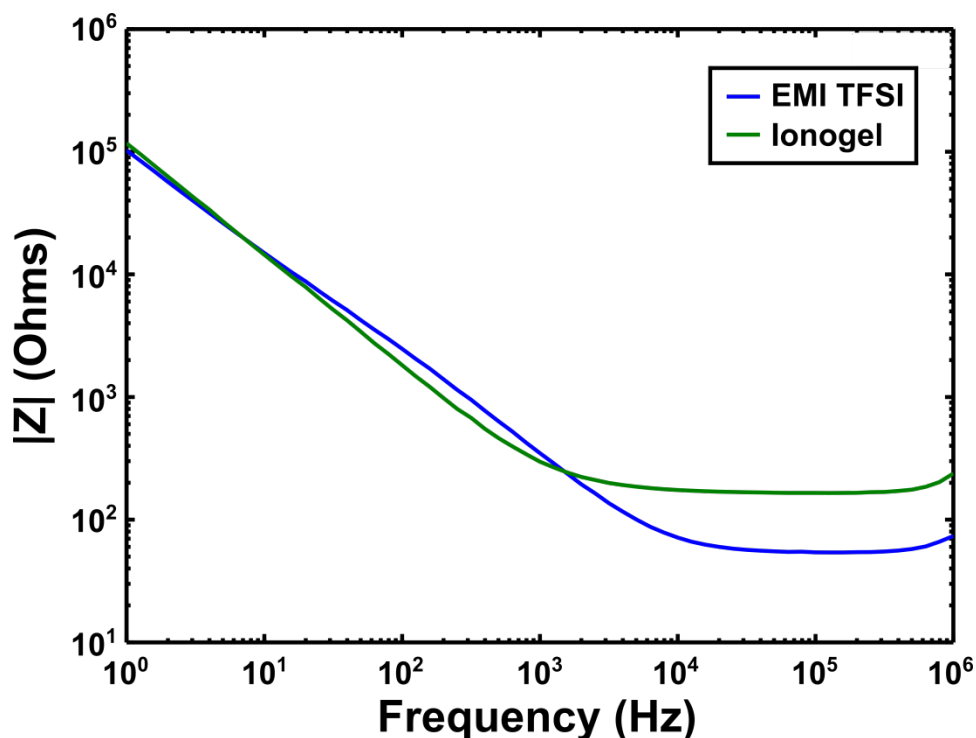


Figure S1. Magnitude of impedance, $|Z|$, versus frequency for EMI TFSI and the ionogel. The larger resistance of the ionogel is apparent at large frequencies. Ionic conductivity values were calculated using the real component of the impedance at $\sim 200 \text{ kHz}$, together with the corresponding electrolyte cross-sectional area and thickness values (for EMI TFSI: $0.31 \text{ cm}^2 \times 0.16 \text{ cm}$; for the ionogel: $0.57 \text{ cm}^2 \times 0.30 \text{ cm}$).