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Nanoporous hybrid electrolytes

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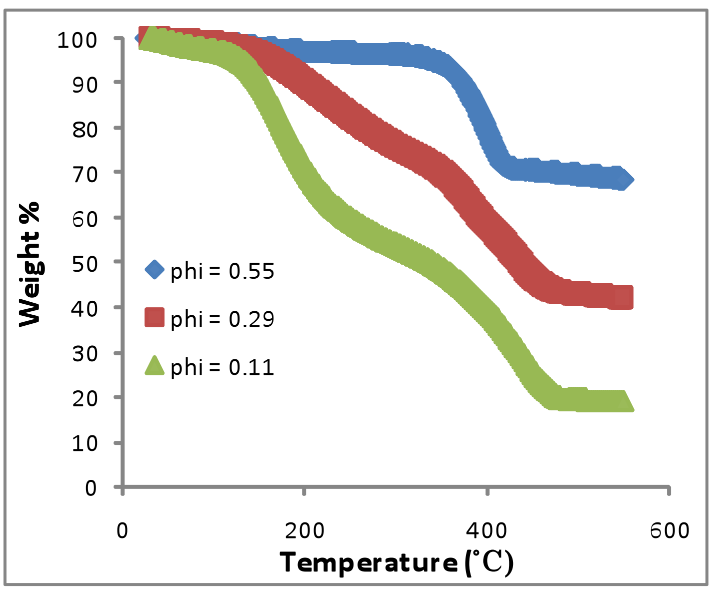
Synthesis

An alkaline stabilized dispersion of silica nanoparticles, Ludox SM-30 (Aldrich) was diluted to 4 wt% particle fraction by addition of aqueous potassium hydroxide solution, pH ~ 10. [Methoxy(polyethyleneoxy)propyl] trimethoxysilane, 90% (Gelest) at a ratio of 0.6 g silane-PEG per 1.0 g silica was added dropwise, while stirring, in three aliquots each separated by heating at 100°C in an oil bath for 1 hour followed by 10-15 minutes of sonication. Following the addition of the final aliquot of silane-PEG, the reaction solution was heated for 6 hours in an oil bath at 100°C. The reaction solution was then poured into wide petri dishes and heated overnight in a convection oven at 70°C to drive off remaining water and complete the silane reaction. The following day, the NOHMs were purified by washing with ethanol 3 times to remove any free silane-PEG, and resuspended in chloroform.

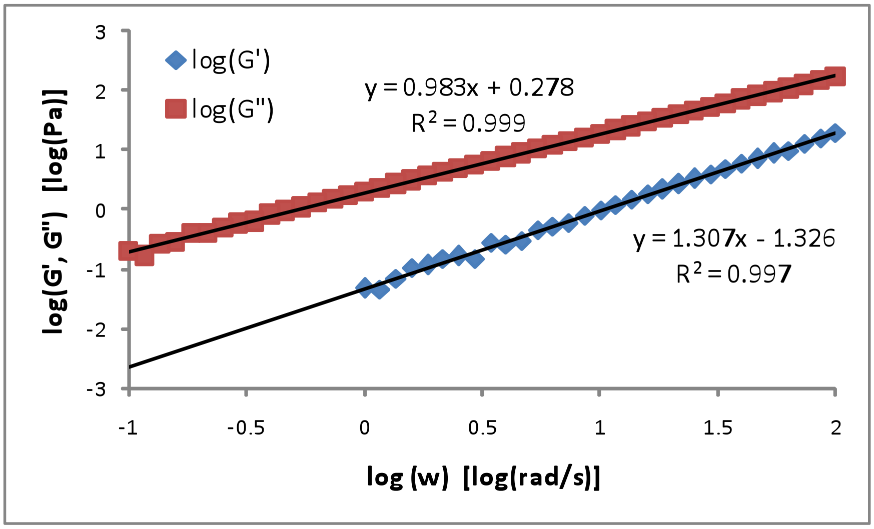
Thermal gravimetric analysis:

TGA plots are shown for pure and plasticized hybrid electrolytes. As shown in the figure, the pure NOHMs electrolyte is thermally stable to above 350°C. The plasticized electrolytes have reduced thermal stability due to the decomposition of PEGDME 250.



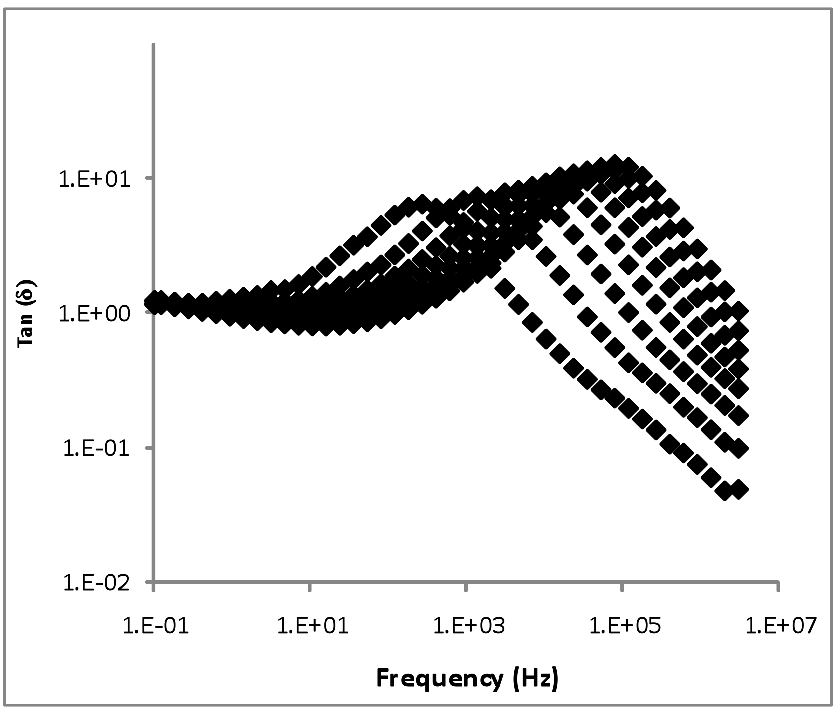
Power law frequency dependence of the moduli:

Example data analysis for a hybrid electrolyte with $\phi = 0.24$. For G', G" ~ ω^m, m is the slope in the applicable fit equation.

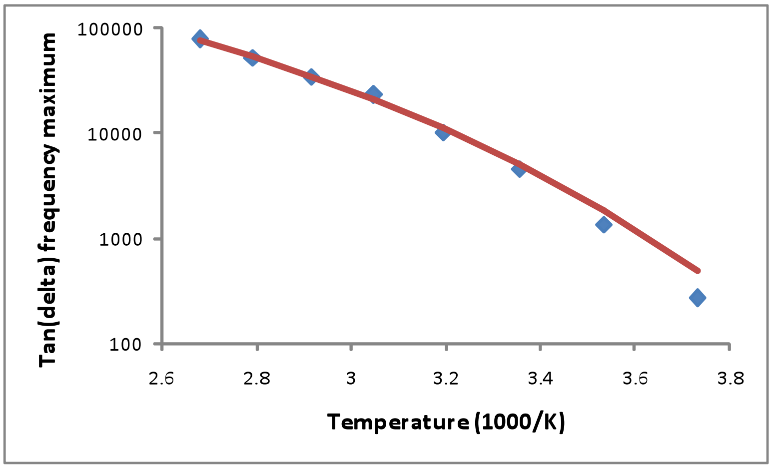


Tan(delta) VFT fit:

Data of tan(δ) vs. frequency at temperatures -5 to 100˚C in 15˚C increments for pure hybrid electrolyte (ϕ = 0.55) as obtained from dielectric spectroscopy. Values of the frequency maximum were recorded for fit to the VFT equation.



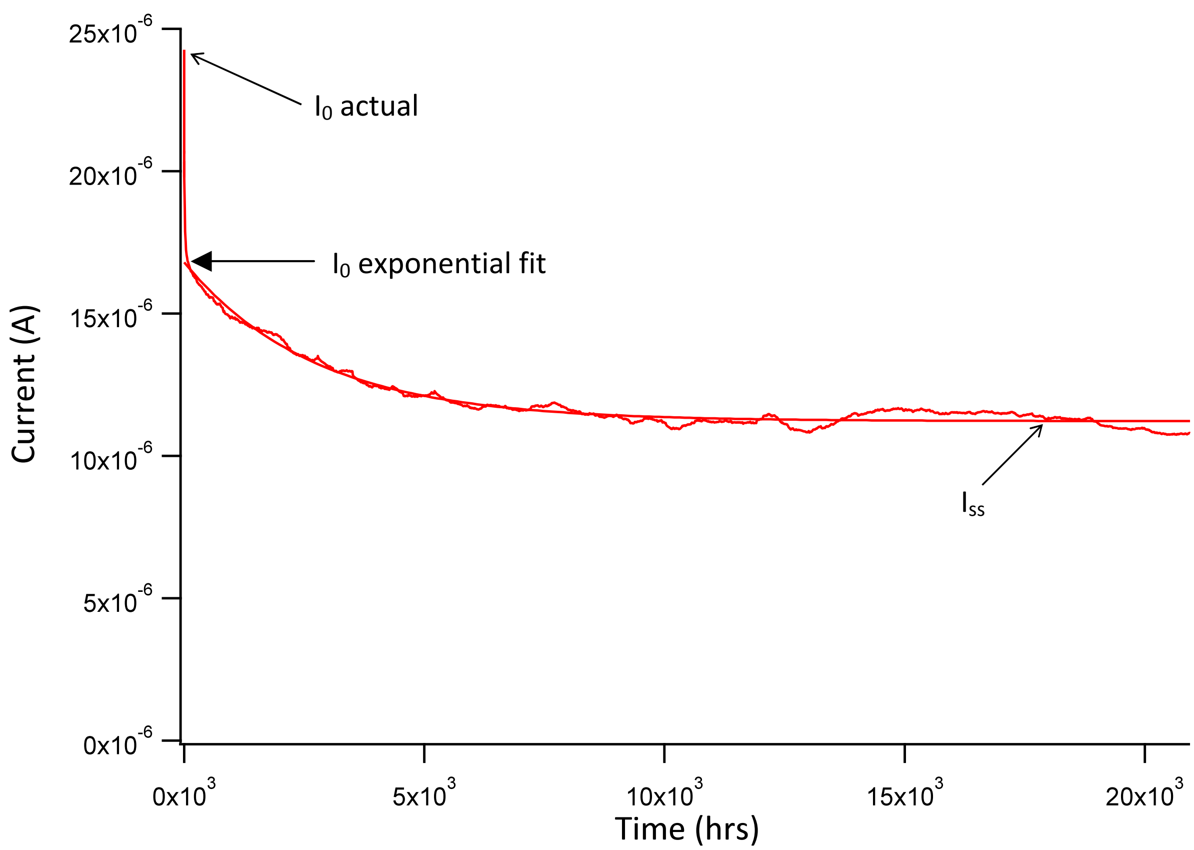
Data points for the frequency maximum of Tan(δ) as obtained from above, fit to VTF equation is line in red. For frequency VTF fit: B = -890 ± 360. Similarly, B = -900 ± 80 for ionic conductivity fit. This suggests that the mechanism for ionic conduction is through semental motion.[ii]



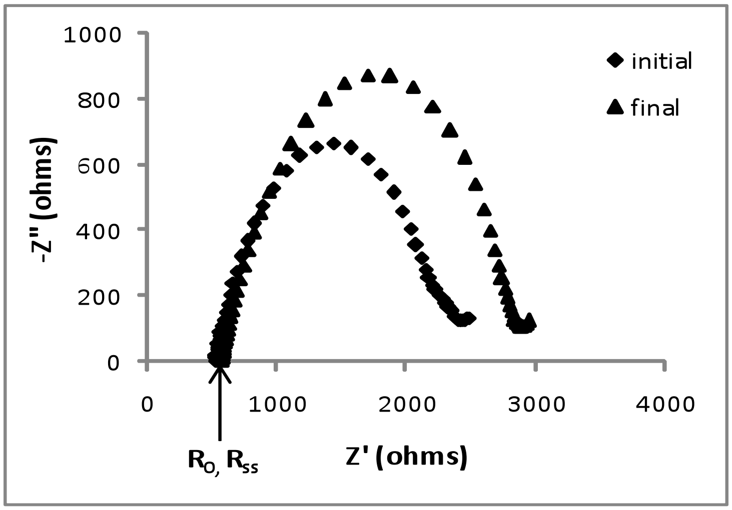
Determination the lithium transference number:

Lithium transference measurements performed on a Li/ electrolyte, ϕ = 0.30 / Li cell using the method proposed by Bruce[iii] and Scrosati[iv] where initial and steady state values of current are found for a symmetric lithium cell undergoing polarization, with corrections from impedance measurements of the interfacial resistance both before and after polarization.

A) Current decay while undergoing a 50 mV polarization. Calculations were performed with the actual I_0 and I_0 determined by fit to an exponential decay function.; I_{ss} was determined by fit to an exponential decay function.



(B). Impedance measurements from 10^4 to 10^{-1} Hz, before and after polarization, to determine interfacial charge transfer resistances, R_o and R_{ss}.



i. Idris, N. H.; Senin, H. B.; Arof, A. K. *Ionics* **2007,** 13, 213-217.

ii. P.G. Bruce, J. Evans, C.A. Vincent, *Solid State Ion.* **1988**, *28–30*, 918.

iii. G.B. Appetecchi, G. Dautzenberg, B. Scrosati, *J. Electrochem. Soc.* **1996**, *143*, 6.