Lisbon flow batteries

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a Schematics of an aqueous organic redox flow battery for grid-scale energy storage. Gray, blue and red spheres refer to K+, Cl-, and SO3- groups, respectively. b Schematic showing the polymer ion-sieving membrane with subnanometer-sized pores that enable fast transport of charge-carrying ions while limiting the crossover of redox active species. Sulfur and oxygen atoms are colored as yellow and red, respectively.

Characterization of the sulfonated polymers was performed using 1H, 13C nuclear magnetic resonance (NMR) and fourier transform infrared spectroscopy (FTIR) spectroscopies, gel permeation chromatography (GPC) and thermogravimetric analysis (TGA) analysis (Supplementary Figs. 3-7 and Supplementary Tables 1 and 2), helium pycnometry and titration measurements, as well as molecular simulations. The resulting polymers possess a range of sulfonate content, associated with ion exchange capacity (IEC) from 0.53-1.86 mmol g-1 (termed as sPIM-SBF-0.53 to sPIM-SBF-1.86) and increased skeletal densities from 1.35 to 1.48 g cm-3, which correlate smoothly to the molar equivalent of TMSCS used in the reaction mixture (Fig. 2c and Supplementary Table 3).

a EIS spectra of K4Fe(CN)6 (1 M)|2,6-DPPAQ (0.4 M) flow battery cells at 100% SOC. b Capacity-voltage profile of the 2nd charging-discharging cycle at a current density of 100 mA cm-2. c Voltage and power density versus current density at ~100% SOC using sPIM-SBF-1.40 and Nafion 115 membrane. d Cycling stability of RFBs using K4Fe(CN)6 (1 M)|2,6-DPPAQ (0.4 M) assembled with sPIM-SBF-1.40 or Nafion 115 membranes at pH = 9 and a current density of 100 mA cm-2. Solid symbols represent normalized discharge capacity and open symbols represent energy efficiency. Normalized discharge capacity data are linearly fitted to derive capacity decay rates. The operating temperature in the glovebox is around 30 ?C.

Nafion(R) 115 membrane (Dupont) was supplied from Sigma-Aldrich. Commercially available chemicals were used without further purification. All reactions using air/moisture sensitive reagents were performed in flame-dried or oven-dried apparatus under a nitrogen atmosphere.

A solution of TMSCS (230 mM) in CDCl3 (100 uL) was added to a solution of 2,2?,3,3?-tetramethoxy-9,9"-spirobifluorene (SBF-OMe, 46 mM) in CDCl3 (400 uL) at 273 K. The sample was then in situ monitored by 1H NMR over a period of 12 h at 278 K. SBF-OMe: 1H NMR (600 MHz,CDCl3): d (ppm) 7.71 (m, 2H, ArH), 7.34 (s, 2H, ArH), 7.32 (m, 2H, ArH), 7.03 (m, 2H, ArH), 6.66 (m,

2H, ArH), 6.23 (s, 2H, ArH), 4.03 (s, 6H, OCH3), 3.64 (s, 6H, OCH3); sSBF-OMe: 1H NMR (600 MHz,CDCl3): d (ppm) 7.89 (br m, 4H, ArH), 7.40 (br s, 2H, ArH), 7.10 (br m, 2H, ArH), 6.21 (br s, 2H, ArH), 4.03 (br s, 6H, OCH3), 3.65 (br s, 6H, OCH3).

Membrane formation was achieved by dissolving PIM-SBF polymer (0.6 g) in CHCl3 (20 mL) or sPIM-SBF polymer (0.7 g) in DMSO (22 mL), which was then centrifuged (12,000 rpm for 5 min) to remove undissolved impurities and poured into a circular glass petri dish (diameter = 9 cm). Membranes were allowed to form by slow solvent evaporation in a desiccator for 48 h at a certain temperature (room temperature for PIM-SBF/CHCl3 casting solution, and 65 ?C for sPIM-SBF/DMSO casting solution). Free standing membranes with thickness around 150 um were peeled off glass petri dishes and cut to needed sizes for further experiments.

The IECs (mequiv/g) of sPIM-SBF membranes were determined by the acid-base titration method33. Membranes were soaked in HCl solution (0.1 M) for 6 h (repeat four times) at room temperature to protonate all sulfonate groups. Protonated membranes were rinsed and immersed in deionized water for 48 h to remove residual HCl. Dry sPIM-SBF membranes in H+ form were immersed in NaCl solution (1 M) for 6 h (repeat four times) to convert H+ form into Na+ form. The exchanged H+ was then titrated with NaOH solution (0.01 M, standardized by 0.010 M potassium hydrogen phthalate) using the phenolphthalein indicator. The values of IEC were determined by the Eq. (1):

where CNaOH is the concentration of NaOH solution, VNaOH is the volume of NaOH solution, and Wd is the weight of dry sPIM-SBF membranes in H+ form. Error bars are standard deviations derived from 3 IEC measurements based on three different samples.