Development of Heterogeneous Ion-Exchange Membrane Fabrication for Vanadium Redox Flow Batteries

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Abstract

Polypropylene (PP)-based heterogeneous anion-exchange membranes (AEMs) for vanadium redox flow batteries (VRFBs) were fabricated by a solvent-free hot-pressing route. A 2^3 factorial design (165/180 °C, 25/50 bar, 5/8 min) and a resin-content series (40–60 wt%) were used to relate processing to structure and properties. Temperature was the dominant variable: the ion-exchange capacity (IEC) ranged from 0.145 to 0.538 meq g^{-1}, and 165 °C, 50 bar, 5 min gave the best balance. All membranes lost < 5% mass in H_2O_2. Raising the resin content to 60 wt% connected the resin particles, lowered the impedance by about 30%, and enabled VRFB charging.

Keywords: anion-exchange membrane; hot pressing; polypropylene; solvent-free fabrication; VRFB

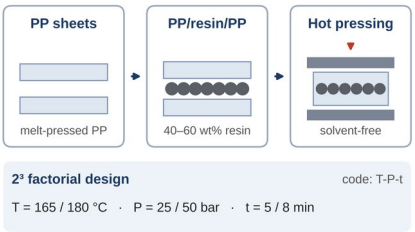
Introduction

Ion-exchange membranes separate the two electrolytes of a VRFB while conducting ions. Heterogeneous membranes, in which resin particles are dispersed in an inert binder, are inexpensive and robust, but their resin domains are often discontinuous, which raises electrical resistance. Most are also produced by solution casting with organic solvents.

Hot pressing consolidates a thermoplastic such as PP with the resin using only heat and pressure. The pressing conditions decide whether molten PP leaves the resin accessible and connected or encapsulates it. **Aim:** to relate processing, structure, and ion transport of solvent-free hot-pressed PP/resin AEMs.

Research Method

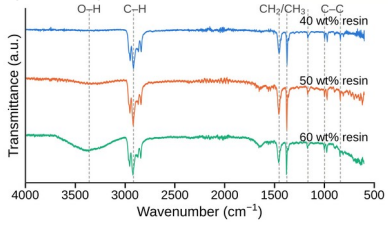
- **Fabrication:** two melt-pressed PP sheets with a quaternary-ammonium resin in between, preheated for 10 min and hot-pressed twice (Picture 1).
- **Design:** 2^3 factorial study at 40 wt% resin; resin content of 40, 50, and 60 wt% at 165 °C, 50 bar, 5 min.
- **Characterization:** FTIR, SEM/EDS, IEC (acid titration), water uptake (WU), fixed-ion concentration (FIC = IEC/WU), water contact angle (WCA), and mass loss in 3 wt% H_2O_2.
- **Transport:** through-plane EIS (10^5–0.1 Hz) and a VRFB (0.1 M V, 20 mA cm^{-2}) benchmarked against IONAC, IONSEP, and FUMASEP; effects tested by ANOVA.



Picture 1. Solvent-free fabrication route and factorial design.

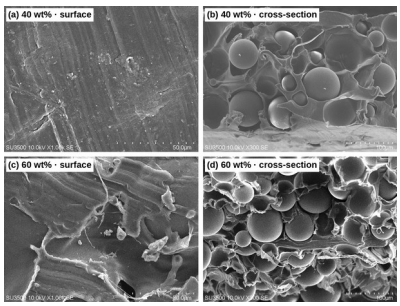
Discussion & Result

Chemical structure. All membranes retained the characteristic PP (C–H, CH_2/CH_3) and resin bands without new peaks, so the membranes formed by physical consolidation rather than chemical reaction. The broad O–H band near 3360 cm^{-1} grew with resin content, reflecting more hydrated resin (Picture 2).



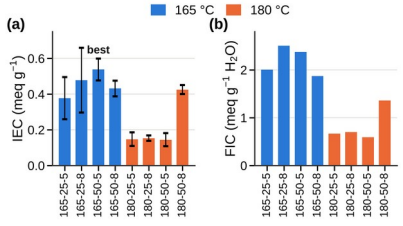
Picture 2. FTIR spectra of membranes with 40–60 wt% resin.

Morphology. At 40 wt%, spherical resin particles were embedded in a continuous PP matrix and rarely exposed at the PP-covered surface. At 60 wt %, the particles were densely packed and touched one another across the thickness (Picture 3).

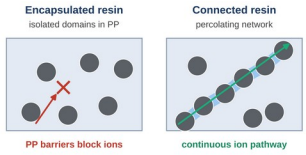


Picture 3. SEM surfaces and cross-sections of membranes with 40 and 60 wt% resin.

Ion-exchange capacity. Temperature significantly affected IEC and FIC (ANOVA, $p < 0.001$). Most membranes pressed at 180 °C had an IEC of only 0.145–0.153 meq g^{-1} despite a WU of 22–24%, suggesting resin encapsulation by the more fluid PP. Membrane 165-50-5 gave the best balance: IEC 0.538 meq g^{-1}, WU 22.6%, and FIC 2.38 meq g^{-1} H_2O (Picture 4). A high IEC alone is not enough: ions must pass through touching, hydrated resin particles (Picture 5).

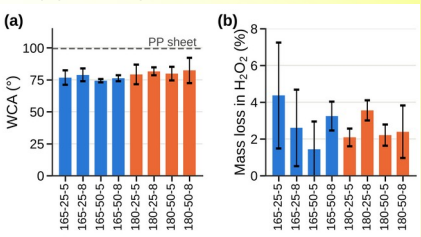


Picture 4. (a) IEC and (b) FIC of the factorial membranes (code: T-P-t).



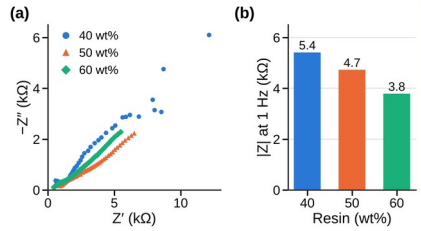
Picture 5. Proposed role of resin connectivity in ion transport.

Surface and stability. WCA values of 74–82°, compared with 99.5° for PP, confirm a PP-dominated surface that became less wettable at 180 °C. Mass loss after 15 days in 3 wt% H_2O_2 stayed below 5%, and was lowest for 165-50-5 (1.44%) (Picture 6).



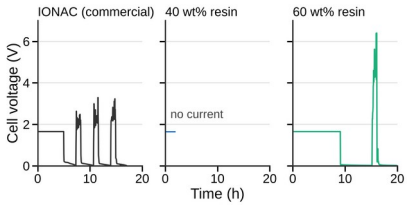
Picture 6. (a) Water contact angle and (b) oxidative mass loss.

Impedance. The spectra showed no closed semicircle, typical of a disordered heterogeneous conductor. The impedance at 1 Hz fell from 5.4 to 3.8 kΩ as the resin content rose from 40 to 60 wt% (Picture 7).



Picture 7. (a) Nyquist plots and (b) |Z| at 1 Hz vs. resin content.

VRFB test. The 40 wt% membrane passed no current during electrolyte preparation. The 50 and 60 wt% membranes could be charged, but needed peak voltages of 9.8 and 6.4 V, compared with ≤ 3.4 V for commercial AEMs, revealing a large ohmic resistance (Picture 8).



Picture 8. VRFB cell-voltage profiles.

Conclusion

- Solvent-free hot pressing produces PP/resin AEMs; temperature is the dominant processing variable.
- 165 °C, 50 bar, 5 min gives the best balance: IEC 0.538 meq g^{-1}, FIC 2.38 meq g^{-1} H_2O, and 1.44% oxidative mass loss.
- Ion transport is limited by resin connectivity rather than by the number of exchange sites; higher resin loading lowers the impedance.
- Next step: continuous, surface-accessible resin pathways (resin loading, particle size, thinner PP layers) for VRFB operation.

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