## **Electronic Supplementary Information**

## High-performance composite membrane based on synergistic main-chain/sidechain proton conduction channels for vanadium redox flow battery

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## Characterization

*Small-/Wide-angle X-ray Scattering* was conducted on Xeuss 2.0 SAXS/WAXS system (Xenocs SA, France). X-ray radiation was launched by Cu K $\alpha$  radiation generator with wavelength = 1.5418 Å at 50 Kv voltage and 0.6 mA current. The SAXS patterns were collected by a semiconductor detector (Pilatus300 K, DECTRIS, Swiss). The membranes were tested under completely hydrated conditions in the atmosphere

 $q = \frac{4\pi \sin \theta}{\lambda}$ 

of nitrogen. The scattering vector q (nm<sup>-1</sup>) was calculated through



**Fig. S1** SAXS profiles of SPEEK membrane (a) and SPEEK/SPPTA composite membranes (b: SPEEK/SPPTA-10; c: SPEEK/SPPTA-20; d: SPEEK/SPPTA-25; e: SPEEK/SPPTA-30).



**Fig. S2** Compared efficiencies (a) and cross-sectional SEM image of Nafion 117 (b and c) and SPEEK/SPPTA-25 (d and e) at 100 mA cm<sup>-2</sup> before and after cycles.



**Fig. S3** Compared FT-IR spectra (a), X-ray patterns (b) and TGA (c) of Nafion 117 and SPEEK/SPPTA-25 membrane before and after cycles.