

Electronic Supplementary Information for

Unveiling the multiscale morphology of chemically stabilized proton exchange membranes for fuel cells by means of Fourier and Real space studies

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ESI-1. Focused Ion Beam-Scanning Electron Microscopy tomography (3D-FIB-SEM)

Movies S2 and S3 display 3D-FIB-SEM stacks of SE and BSE images (500 images for a volume of 3x3x2.5 μm^3) obtained on the sample HyM-32%-PT. These stacks of images confirm the AFM observation of SG aggregates being formed by smaller particles. As the SG phase contains silicon atoms, absent in the sPEEK phase, the brighter the area on the BSE image, the more it contains silicon (higher atomic number: 14 for silicon vs. 8 for oxygen and 6 for carbon), and therefore SG.

Links to download the movies:

S2 : <https://drive.google.com/file/d/1nVqWfD7s6gPnVRM63MVaQK1NanB4YSVr/view?usp=sharing>

S3 : https://drive.google.com/file/d/1aJfnV1hVrd8gnVj_cEY9OY5AggvJxqLY/view?usp=sharing

ESI-2. Wide Angle X-ray Scattering (WAXS)

Fig.S1 displays the WAXS data obtained for a pristine sPEEK membrane (sPEEK_NS_PT), an *ex-situ* synthesized SG sample and HyM-32%-PT. The hybrid membrane pattern can be mainly considered as the sum of the patterns of the membrane and SG components indicating the absence of structural modifications (of the sPEEK by the growing SG phase, and of the SG phase when it grows inside the sPEEK instead of *ex-situ*) at this scale (from a few Å up to a few nm).

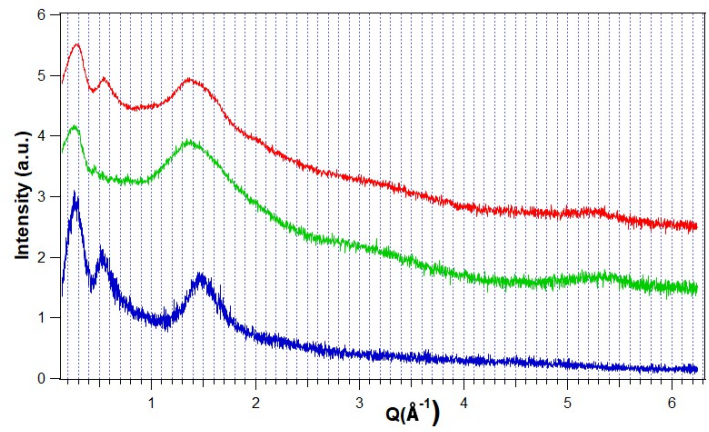


Fig. S1. WAXS data obtained with the SPEEK membrane (SPEEK_NS_PT) (green), the ex-situ synthesized sol-gel phase (blue) and HyM-32%-PT (red).