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Ptychographic X-ray computed tomography of porous membranes with nanoscale resolution

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Background Incl. Aims
3D images of porous materials in high resolution have been so far obtained using transmission electron tomography or focused ion beam coupled with scanning electron microscopy. These methods require ultra-vacuum conditions, and only a small volume of the sample is visualized. Here, we demonstrate the application of ptychographic x-ray computed tomography for visualizing soft matter with a resolution of 26 nm over large fields of view. We showcase the technique's capabilities by imaging polymer hollow fiber porous membranes.

Porous materials have essential functions in nature, whether for water and nutrient transport in plants and other biological systems or for the storage of oil and water in rock reservoirs. Synthetic polymeric materials are key for chromatography and membrane separation processes. Their application is well-established in water desalination, hemodialysis, gas separation, and is expanding in nanofiltration for chemical separations. The advantages of membrane technology are low energy requirement, and low carbon footprint combined with a compact design that allows packing a large membrane area within a small volume. Effective membranes have a complex porous structure to secure selectivity, mechanical stability, and fast transport characteristics. Porosity and interconnectivity determine the membrane performance.

Thanks to the high-penetration depth of the X-ray beam, we visualize the 3D complex porous structure of polyetherimide ultrafiltration hollow fibers in a non-destructive manner and obtain quantitative information about pore size distribution and pore network interconnectivity across the whole membrane wall. The non-destructive nature of this method, coupled with its ability to image samples without requiring modification or a high vacuum environment, makes it valuable in the fields of porous- and nano-material sciences, enabling imaging under different environmental conditions.

Methods
A highly coherent x-ray beam produced by SIRIUS, the 4th generation synchrotron source inaugurated in 2020 in Campinas, Sao Paulo, Brazil, was used at a dedicated beamline for x-ray imaging - Cateretê. Ptychographic x-ray computed tomography (PXCT) is a lensless coherent x-ray technique, where the specimen is raster scanned in two dimensions by a coherent x-ray beam defined by a pinhole, and diffraction patterns in the far-field are recorded at each position. The sample is rotated and the 2D scans are collected at different projections to obtain a 3D dataset for reconstructions.

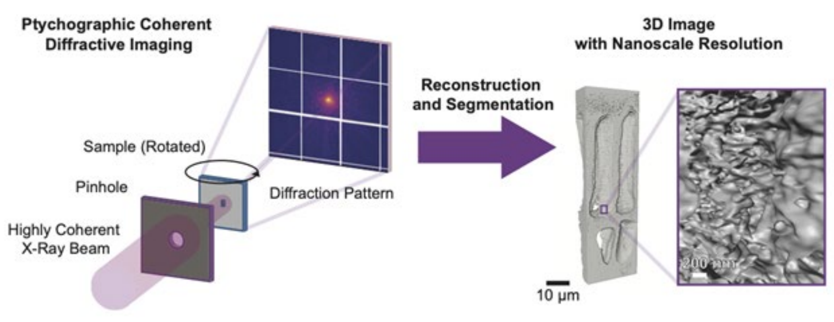
Results

The high-performance computer reconstruction of the collected 2D projections resulted in detailed 3D images of the hollow fibers, with a resolution of 26 nm and a voxel size of 23 nm × 23 nm × 23 nm. Such a detailed model contains not only visual but also quantitative information, and it was used to evaluate the porosity gradient across the membrane walls for hollow fiber membranes. Apart from the porosity, we were able to study the evolution of the diameter size of every detected pore channel across the membrane thickness within the resolution limits (26 nm diameter), to evaluate the pore size distribution and changes across the entire membrane. We could also provide a comprehensive analysis of the interconnectivity of the pores.

Conclusions

The highly coherent X-ray beam generated by the 4th generation synchrotron source allows for the non-destructive analysis via PXCT with nanoscale resolution. The reconstruction of the X-ray coherent diffraction patterns from different projection angles, not only facilitated the visualization of complex porous morphology throughout the entire membrane wall volume but also provided a detailed 3D quantitative model of the porous network. The presented PXCT method is not limited to the measurements of porous structures but can be employed for resolving the nanostructure details in large samples within soft matter. Furthermore, thanks to the high-penetration depth of X-rays, PXCT allows for the analysis of the samples where the sectioning for electron microscopy methods is not feasible. The lack of vacuum requirements and the absence of sample modification, such as heavy metal staining, enable in situ analysis of samples in different environmental conditions. These advantages, coupled with the non-destructive nature of this technique, make it highly applicable in material science and biology for the understanding of complex porous- and nanomaterials offering nanometer-scale resolutions for large sample volumes and providing valuable quantitative information.

Graphic:



Keywords:

X-Ray Tomography, Ptychography, Polymer Membrane

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