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In-situ 3D X-ray Tomography and Analysis of Reverse Osmosis Membranes Under Compaction

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The need for attention to freshwater protection and management is rising as water scarcity becomes more prevalent in the coming years. The water treatment process of reverse osmosis (RO) removes contaminants from water by using pressures to force water molecules through a semipermeable multi-layer membrane. Current seawater RO (SWRO) membranes designed for thermal desalination are energy intensive and unable to withstand high pressures. The development of high-pressure reverse osmosis (HPRO) membranes can result in a more energy-efficient and effective desalination process. [1] To accomplish this task, the HPRO membranes must maintain their microstructural properties at high pressures up to 200 bar. Normally, membrane performance is studied using methods like *molecular dynamics simulation* and *compaction modeling and prediction* which are time-consuming and prone to error. [2] Here we showcase a developed workflow using non-destructive X-ray computed tomography (XCT) and AI-segmentation to compare SWRO and HPRO membrane microstructures and analyze performance under high pressures.

A *Zeiss Xradia 520 Micro-CT* with a *Deben CT-5000* in-situ stage was utilized to compare traditional SWRO and HPRO membranes by compacting them to simulate water flow. The membranes were mounted in an in-situ stage and imaged in the micro-CT. An unloaded, control scan was conducted on both membranes initially, to provide a baseline for comparison. Multiple pressures up to a maximum of 200 bar were applied over a 3mm diameter membrane and imaged at a resolution of 2µm. Typically, the low density of the membranes introduces challenges when attempting to perform an XCT image, resulting in poor signal to noise and unusable data. To overcome this challenge and fully resolve the microstructures of the membranes, an iterative reconstruction method was applied. Compared to the traditional reconstruction technique, known as filtered back projection, this iterative method greatly decreases noise and enhances the contrast of the dataset. Furthermore, the iterative reconstruction of 3D tomography enables us to scan at 1201 projections rather than 2401 projections, reducing the scan time by 50%. [3] This reduction in scan time is crucial to decrease motion artifacts that can arise from the high pressures placed onto the membranes. As a result, we were able to successfully obtain high quality XCT data of the membranes and microstructures. Figure 1 shows the comparison between traditional filtered back projection reconstruction and iterative reconstruction on a slice from the same membrane's 3D tomography.

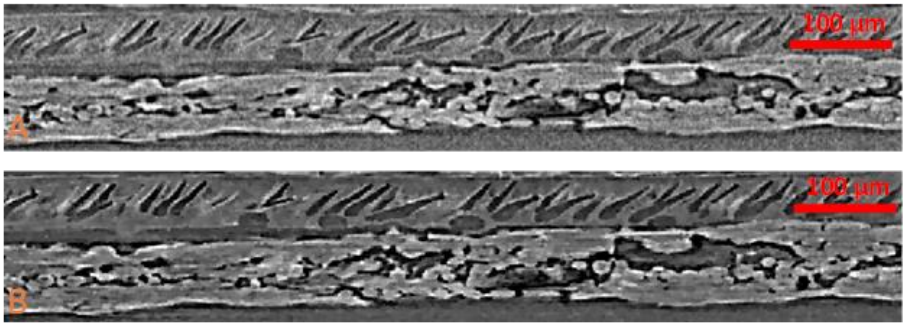

Figure 1. (a) Filtered back-projection reconstruction of in-situ XCT of HPRO membrane (b) Iterative reconstruction of in-situ XCT of HPRO membrane.

To quantify the microstructural properties in each layer of the membranes, an AI-segmentation method was utilized. [4] Conventional methods such as thresholding are unable to produce reliable results due to challenges in separating pores and material in each layer. This is because the low density of the membranes results in greyscale values are close to one another. Also, greyscale values vary from slice to slice due to imaging artifacts inherent to XCT. The mentioned challenges make automated image analysis very difficult. Manual methods are time consuming, human-dependent, and prone to error, eliminating the possibility of a comparative study. The AI-segmentation method allows for a more efficient and accurate segmentation by implementing a machine learning algorithm on a sub-dataset of slices from the 3D tomography. Those slices are then trained to their respective labels in each layer. [5] The AI-algorithm then uses the trained labels to apply the segmentation over the full 3D dataset of SWRO and HPRO membranes before and after compaction. Figure 2 shows the resulting segmentation on the HPRO membrane before compaction.

Figure 2. AI-based segmentation on in-situ 3D tomography of HPRO membrane unloaded (0 bar).

In order to validate this workflow, quantifications such as porosity, thickness, tortuosity and permeability were conducted on the segmented datasets to compare performance before and after compaction. After compaction, the expected changes such as, reduced porosity and increased tortuosity were observed, indicating the HPRO membranes could withstand higher pressures compared to SWRO membranes. With the use of the iterative reconstruction and AI-segmentation processes, it is possible to resolve features in the membrane samples and quantify their performance under pressure. These results support that the in-situ X-ray microscopy and AI-segmentation workflow offers a streamlined way to carry out high resolution 3D characterization of complex samples.

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