

MEMBRANES

Nanoscale control of internal inhomogeneity enhances water transport in desalination membranes

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Biological membranes can achieve remarkably high permeabilities, while maintaining ideal selectivities, by relying on well-defined internal nanoscale structures in the form of membrane proteins. Here, we apply such design strategies to desalination membranes. A series of polyamide desalination membranes—which were synthesized in an industrial-scale manufacturing line and varied in processing conditions but retained similar chemical compositions—show increasing water permeability and active layer thickness with constant sodium chloride selectivity. Transmission electron microscopy measurements enabled us to determine nanoscale three-dimensional polyamide density maps and predict water permeability with zero adjustable parameters. Density fluctuations are detrimental to water transport, which makes systematic control over nanoscale polyamide inhomogeneity a key route to maximizing water permeability without sacrificing salt selectivity in desalination membranes.

Nearly 80% of worldwide fresh water is used for agriculture, livestock, and energy applications, which places substantial stress on existing water sources in both developed and developing countries (1, 2). Technologies such as membrane filtration, distillation, and ion exchange are extensively used to purify water (3, 4); nonetheless, the energy requirement to remove dissolved solutes, particularly salt, from water remains high.

Reverse osmosis (RO) (5) occupies a 66% share of the global desalination capacity and produces nearly 21 billion gallons of water per day (6). It is also playing an increasingly important role in recycling and recovering fresh water from wastewater and other waste streams for both human and industrial use (7, 8). Recent progress in RO membrane synthesis has yielded methodologies to manufacture highly permeable desalination membranes by controlling the internal morphology, thickness, and feed surface area of the fully aromatic polyamide (PA) active layer (9–12). It is not clear, however, how the resulting nanoscale PA morphology is linked to the performance observed in such membranes.

We describe a methodology to quantify the effect of three-dimensional (3D) nanoscale variations in polymer mass on water transport

within the PA active layer for a series of four RO membranes (PA1 to PA4). The PA films were synthesized by a conventional interfacial polymerization reaction between aqueous diamine and organic acid chloride solutions directly on a porous polysulfone support membrane (13, 14) conducted in a commercial pilot-scale manufacturing line (see materials and methods for synthesis details). The performance of the synthesized membranes was evaluated using cross-flow filtration (table S1). To isolate morphological influences on water transport properties, the differences in chemical composition between the membranes used were minimized, as previously described (13). Fourier transform infrared spectroscopy profiles (fig. S1) confirm a nearly constant carboxylic acid-to-amide ratio. Through a combination of electron tomography, energy-filtered transmission electron microscopy, and solution-diffusion simulations, we find that nanoscale variations in density are detrimental to water transport in these membranes and that controlling these density fluctuations is crucial to maximize performance in RO membranes (15, 16).

Transmission electron microscopy (TEM) has not been able to quantitatively link the PA microstructure with desalination performance (17–19). When imaging in scanning TEM (STEM) mode using a high-angle annular dark-field (HAADF) detector, images are formed by Z-contrast (20), where, for a single-component system, the pixel intensity is directly related to the sampled mass. By extension, for an isolated PA film, the pixel intensity of a HAADF-STEM image is a function of the sample thickness, density, and pixel size. To decouple PA thickness and density in the electron microscope, 3D reconstructions of the nanoscale PA morphology are necessary. We achieve this through HAADF-STEM tomography, where a tilt series is aligned to create a 3D model that describes

the nanoscale surface and internal PA morphology (details in the supplementary materials; Fig. 1, A and B; figs. S2 to S5; and movies S1 to S4). Quantification of 3D models reveals that the PA void fraction and surface area are consistent with analysis of similar commercial RO membranes (fig. S6) (21).

High-resolution HAADF-STEM tomography decouples PA density and thickness, which allows for the determination of nanoscale 3D distributions of each of these parameters independently. Although average values of relevant membrane properties are commonly used to estimate membrane transport rates, nanoscale distributions of mass likely govern transport through RO membranes (evident in Fig. 1, C and D). Variations in membrane resistance and water flux would therefore arise from a combination of nanoscale variations in PA thickness and density. We can use a combination of energy-filtered TEM and HAADF-STEM tomography to map how the nanoscale inhomogeneity in the density varies within PA films and how it relates to variations in membrane thickness. As described in detail in the supplementary materials, we convert the 3D nanoscale intensity distributions (Fig. 1, C and D) to nanoscale distributions of density (ρ), from which we extract the water diffusivity (D_w) within PA films (details in the supplementary materials; figs. S7 to S22; tables S2 and S3; and Fig. 1, E to J). We calculate the diffusivity from the density in a solution-diffusion formalism through the fractional free volume (FFV) by estimating the occupied volume from the maximum polymer density that we measured in our tomograms. This is effectively an excess free volume (22), where the occupied volume from the maximum density in 3D reconstructions is similar to reported values (23) and estimates from the Bondi method (24). We thus use the term scaled fractional free volume (sFFV) for values obtained using electron tomography to denote the introduction of this approach for estimating free volume. The color gradients under each curve for the density, sFFV, and water diffusivity shown in Fig. 1, E to J, serve as an absolute scale for the cross section in Fig. 1, C and D (figs. S19, S20, and S22 for PA2 and PA3).

A precursor for determining nanoscale 3D PA inhomogeneities is the measurement of the average values of PA density (ρ_{avg}), the average sFFV ($sFFV_{avg}$), and the average diffusion coefficient of water ($D_{w,avg}$). In short, by accounting for the elastic and inelastic scattering components of a TEM image (Fig. 2, A to C), we can obtain the mean free path of electrons (details in the supplementary materials, figs. S7 to S11, and table S2). The mean free path can be used to determine ρ_{avg} and, by extension, $sFFV_{avg}$ and $D_{w,avg}$ in PA films (details in the supplementary materials; Fig. 2, D and E; and fig. S13). In the series of membranes we

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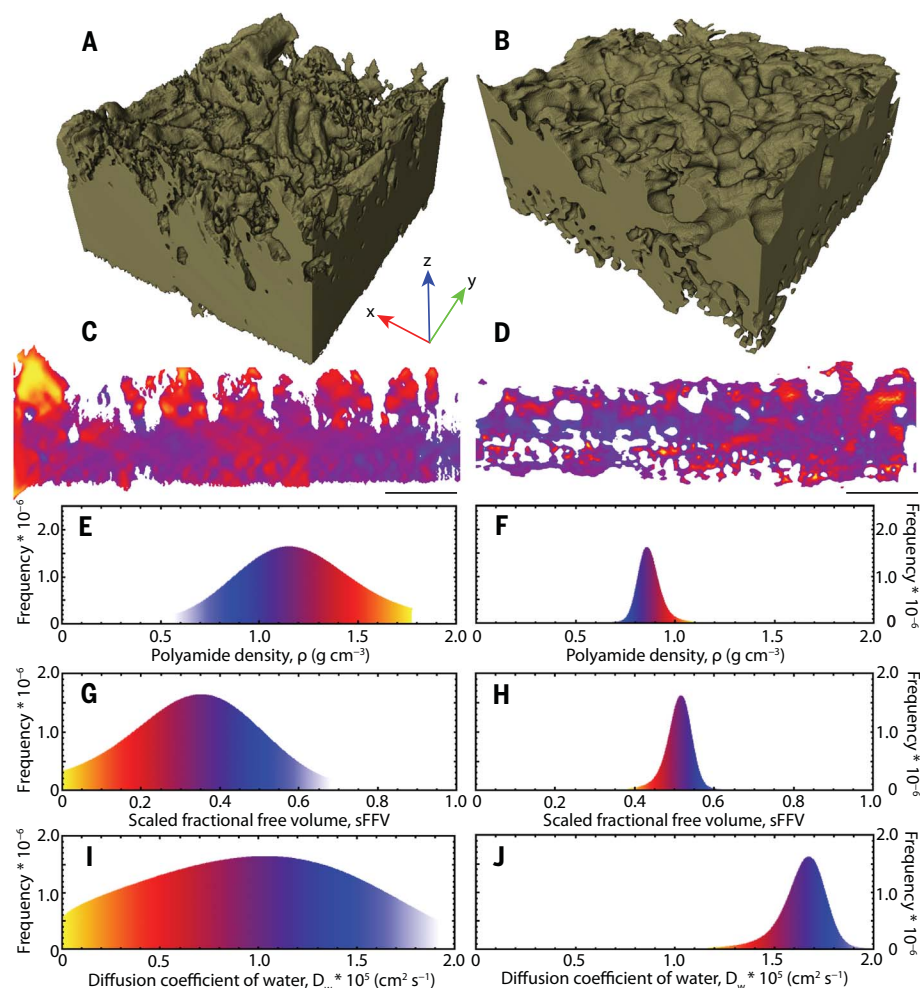


Fig. 1. Quantifying the 3D nanoscale inhomogeneity of PA RO membranes through the combination of energy-filtered TEM and electron tomography. (A and B) 3D isosurfaces of the PA1 (A) and PA4 (B) membranes. (C to J) 12-Å-thick xz plane with colored voxels of PA1 (C) and PA4 (D) corresponding to colored gradients under the density [(E) and (F)], sFFV [(G) and (H)], and diffusion coefficient [(I) and (J)] of water histograms for the PA1 and PA4 membranes, respectively. (A), (C), (E), (G), and (I) show data for PA1; (B), (D), (F), (H), and (J) show data for PA4. All studied membranes show internal nanoscale inhomogeneity. Length axis arrows and scale bars are 200 nm. Histograms were obtained from reconstructions of PA films with $>10^8$ voxels.

tested, the water permeance increased from 6.39 ± 0.22 to 8.36 ± 0.15 liters m^{-2} hour^{-1} bar^{-1} (LMH/bar) and was correlated to the ρ_{avg} decrease from 1.15 ± 0.14 to 0.86 ± 0.09 g cm^{-3} (Fig. 2F). These average density values are in agreement with literature-reported bulk PA density values (25) and are consistent with an ~ 4 - to 4.6 -Å spacing between chains, assuming liquid-like packing (with aligned chain backbones for simplicity). Further, over the same permeance increase, sFFV_{avg} increases from 0.35 ± 0.04 to 0.52 ± 0.05 (Fig. 2G), which indicates that increases in angstrom-scale free volume have a positive correlation with water flux. The large sFFV values are consistent with FFV predictions for glassy polymers and indicate that the PA free volume elements are likely interconnected (26). $D_{\text{w,avg}}$ values were

obtained from a combination of free volume theory (27) and a compilation of D_{w} versus $1/\text{sFFV}$ data from molecular dynamics simulations (fig. S21). The expected overall trend from the solution-diffusion model (26), commonly invoked to describe transport in RO membranes, was followed as $D_{\text{w,avg}}$ increases from 1.03 ± 0.02 to $1.67 \pm 0.04 \times 10^{-5}$ $\text{cm}^2 \text{s}^{-1}$ and as the water permeance increases from 6.39 ± 0.22 to 8.36 ± 0.15 LMH/bar (Fig. 2H). $D_{\text{w,avg}}$ values were consistent with values for PAs measured using quasi-elastic neutron scattering (15). These results indicate that ρ_{avg} , sFFV_{avg} , and $D_{\text{w,avg}}$ can be determined directly from TEM measurements.

The progressively narrowing D_{w} distributions from PA1 to PA4 as water permeance increases indicate that local distributions

in mass affect water transport. Nevertheless, water transport properties cannot be predicted exclusively from these distributions because the nanoscale water diffusivity distributions in Fig. 1, I and J, do not account for variations in membrane resistance. The spatial arrangement of localized membrane resistance variations plays a crucial role in determining diffusion pathways. Water molecules would be more likely to diffuse through a PA region of low thickness and density compared with a nearby thick and dense region—i.e., water transport would take the path of least resistance. Further, these variations in resistance would cause distributions in flow, causing flux hot spots (28), which cannot be accounted for purely on the basis of local or simply averaged D_{w} . The inability of average values to reliably predict transport properties is further highlighted by water flux calculations based on the average values reported in Fig. 2, E and H (i.e., for a PA film of uniform density and thickness), and the solution-diffusion model of water permeating through a nonporous membrane. The predicted water flux, based solely on average values, indicates decreasing water flux with increasing average thickness (table S4), which is opposite to the observed trend. The observed trend of increasing water flux with increasing thickness in this series of membranes (13) is counterintuitive and differs from previously reported results (29).

To predict transport properties, we calculate water diffusion through 3D models that show how thickness and D_{w} vary locally, which are obtained from the combination of energy-filtered TEM and electron tomography. We solve for the nanoscale variations in water transport by applying Fick's law with zero adjustable parameters at every 1.7-nm^3 voxel, totaling >100 million voxels per 3D model. We ignore frame-of-reference effects (30) given that the water content is <15 vol % (21, 31). This allows us to connect directly with simulations that estimate diffusion coefficients where frame-of-reference effects are also neglected (fig. S21). The boundary conditions are the concentrations of water at both the membrane feed and permeate surfaces determined by means of the solution-diffusion model (see calculation and details in the supplementary materials) (31). As a result, water diffusion pathways through PA films can be determined (Fig. 3) where the effect of nanoscale PA morphology on 3D water transport can be visualized. Light gray regions correspond to regions of ultralow water diffusivity within the membrane ($D_{\text{w}} < 5 \times 10^{-6}$ $\text{cm}^2 \text{s}^{-1}$), which correspond to regions of high PA density and low sFFV. Dark gray regions correspond to water diffusivity between 1.2 and 1.5×10^{-5} $\text{cm}^2 \text{s}^{-1}$. The regions of greatest resistance are near the PA top surface, which emphasizes the importance of PA surface area on water transport

rates. Water diffusion pathways show variations in the x and y directions to avoid these areas, which indicates that water flow in the commonly seen surface polyps is low. Using the flow maps, we can reliably predict water permeability with zero adjustable parameters (Fig. 4).

The insets of Fig. 4 denote xy planes corresponding to predicted water flux distributions ($J_{w,p}$) into and out of the reconstructed

PA volume as a result of density and thickness inhomogeneities for the PA1 and PA4 membranes (PA2 and PA3 are shown in figs. S23 and S24). Although all membranes show some local inhomogeneity in water flux, we find that the highest flux membrane (PA4) minimizes low-flow regions. A comparison of the flow distributions at several xy planes within the membrane and on each surface reveals evidence of lateral water transport (x

and y directions), which indicates that the nanoscale PA morphology affects water transport in all three dimensions (Fig. 4, inset, and figs. S25 and S26). Using the calculated flow maps, we can reliably evaluate the predicted water permeability, $P_{w,p}$, showing qualitative agreement with measured water permeabilities from cross-flow filtration testing, $P_{w,m}$ (Fig. 4). Small deviations between $P_{w,p}$ and $P_{w,m}$ could result from unaccounted effects introduced by the polysulfone support layer (9). $P_{w,p}$ values are upward of 27% greater than water permeability predicted from a smooth PA film with a single homogeneous permeability (with the exception of PA3), which indicates that nanoscale internal inhomogeneities have a large effect on water transport in thick membranes (table S5).

The most permeable membrane (PA4) has the lowest average density and narrowest density distribution, which suggests that highly permeable membranes minimize mass fluctuations that suppress water permeability, thereby maximizing overall permeability while maintaining selectivity. This is consistent with strategies to maximize both permeability and selectivity in gas separation membranes (32). Confining density values to a narrow distribution just below the boundary where the solute selectivity trade-off would be compromised could provide the highest possible water permeability for a desired selectivity. This would likely be in the form of a uniform density and resistance-selective region within the PA film, previously discovered to exist primarily near the PA feed surface (21) and illustrated by data shown for PA4 in Fig. 1 and density-thickness profiles in fig. S27. PA1 to PA3 have broader

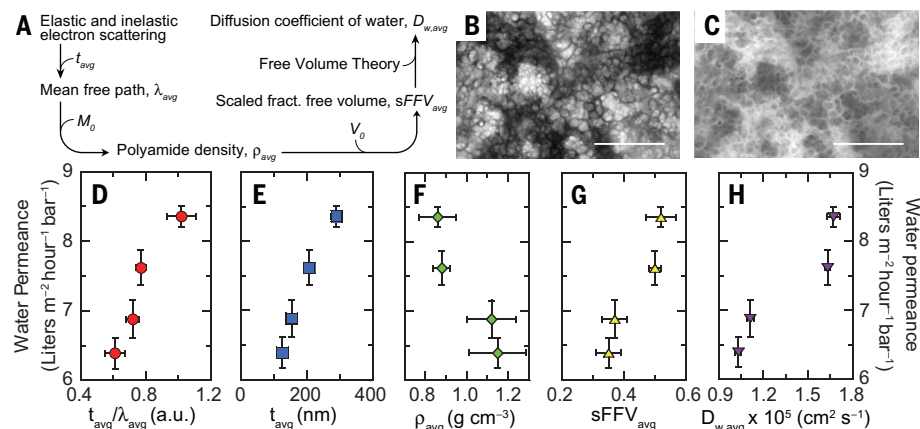


Fig. 2. Determining the average density, free volume, and diffusion coefficient of water in PA films using energy-filtered TEM. (A) Flow chart of the process to determine the average diffusion coefficient of water in PA films. (B and C) Selected areas from a zero-loss (B) and composite thickness map image (C) of the PA4 membrane. Scale bars, 500 nm. (D and E) Water permeance as a function of average thickness map intensity, $t_{\text{avg}}/\lambda_{\text{avg}}$, (D) determined from (B) and (C), and average thickness, t_{avg} (E) (measured by means of ellipsometry). (F to H) Water permeance as a function of average PA density, ρ_{avg} , (F) determined from (D) and (E); PA monomer molecular weight, M_0 , (G) sFFV_{avg} determined from (F); and PA monomer specific occupied volume, V_0 , and diffusion coefficient of water, $D_{w,\text{avg}}$, (H) determined from (G) and free volume theory for PA1 to PA4 membranes (details in the supplementary materials). Error bars are standard deviations with $N = 5$ for PA1 to PA3 and $N = 6$ for PA4. a.u., arbitrary units.

Fig. 3. Calculating water transport through 3D models obtained from energy-filtered TEM and electron tomography. (A to D) Perspective views [(A) and (B)] and cross sections [(C) and (D)] of the water diffusion pathways through the PA1 [(A) and (C)] and PA4 [(B) and (D)] membranes.

Gray areas in (A) represent regions of ultralow water diffusivity ($D_w < 5 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$) corresponding to regions of high PA density ($\rho > 1.35 \text{ g cm}^{-3}$) and low sFFV (sFFV < 0.24). Gray regions in (B) correspond to water diffusivity between 1.2 and $1.5 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$. The streamlines are colored based on the local flux values in liters per square meter per hour. Larger regions of low water diffusivity in PA1 result in lateral water diffusion to avoid these high-resistance regions. Water diffusion pathways in PA4 show less lateral movement within the membrane, indicating smaller regions of high membrane resistance. Axis labels are scaled to 200 nm.

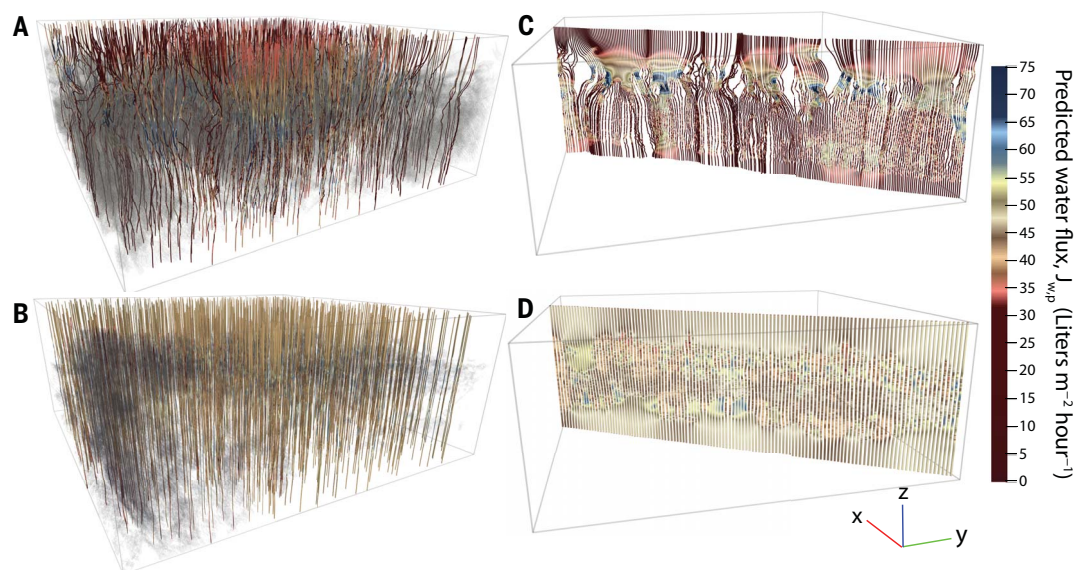
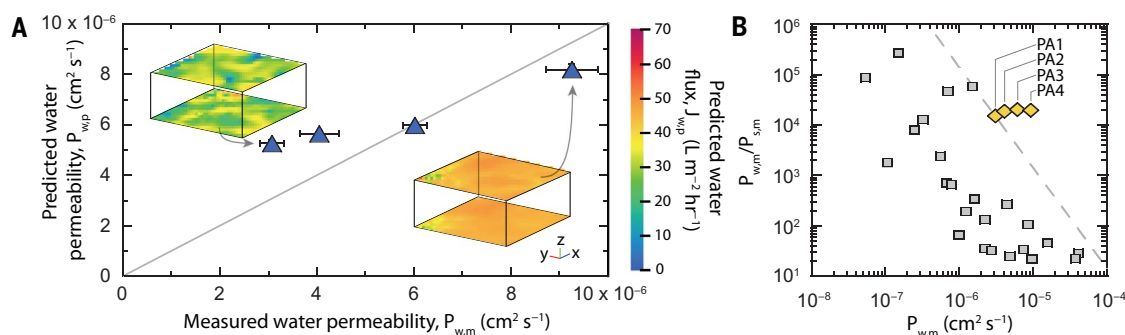


Fig. 4. Nanoscale water transport calculations predict water permeability with zero adjustable parameters and comparison of membrane performance with state-of-the-art membranes. (A) Predicted water flux ($J_{w,p}$) maps detail the flow distribution for the PA1 and PA4 membranes (insets). By application of Fick's law, water permeability

can be predicted ($P_{w,p}$) and compared with measured water permeability ($P_{w,m}$). Flow distributions arise from nanoscale variations in PA resistance, where the PA1 flow distribution shows large inhomogeneities because of its broader density distribution. Conversely, the water flux distribution of PA4 is more homogeneous because of its narrower density distribution. Gray line serves as a guide to the eye. (B) Measured water-NaCl selectivity versus measured water permeability for desalination membranes used in this study compared with reported membranes. Yellow markers indicate PA1 to PA4 membranes and gray squares represent literature values. Desalination membrane data and upper bound line are from (31).



density distributions in addition to higher average values and, thus, larger regions of low water diffusivity. We propose that a minimum average PA density exists that enables monovalent salt selectivity for suitable desalination specifications [e.g., a salt-to-water selectivity of 1:10,000 for seawater desalination (31)]. A membrane with an even narrower density distribution than PA4 approaching this threshold value would result in the effective water permeability approaching the true upper limit of fully aromatic PA RO membranes (for a given NaCl permeability). The synthesized RO membranes are near or above the upper-bound trade-off line for desalination membranes (Fig. 4B), which indicates that the open structure of PA4 already approaches this hypothesized upper limit for water permeability at brackish water salt retention levels (13).

We evaluated the generality of this approach with a similar analysis of a polyethersulfone ultrafiltration membrane used for virus filtration in downstream processing in the biopharmaceutical industry. 3D reconstructions reveal tortuous open pathways for water transport (fig. S28), and we again calculate flow properties. Despite different transport mechanisms compared with that of flow through dense PA films, an accurate effective diffusion coefficient and water flux can be obtained by accounting for the 3D pore network (see the supplementary materials).

The above methodology quantifies structure-property relationships for membranes that exceed literature-reported upper bounds of desalination performance, and it takes a step toward understanding water diffusion mechanisms and predicting transport rates. We demonstrate that the combination of energy-filtered TEM and electron tomography—i.e., multimodal electron microscopy—is a key tool to create predictive correlations between morphology and water transport for high-performance RO membranes. These cor-

relations can be extended to other molecular separation and polymeric systems to improve design strategies for various applications, including gas and hydrocarbon separations, carbon capture, blue energy production, and desalination.

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SUPPLEMENTARY MATERIALS

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Materials and Methods
Supplementary Text
Figs. S1 to S28
Tables S1 to S5
References (34–54)
Movies S1 to S4

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Finding the path to better desalination

Polyamide membranes have been used in large-scale desalination for decades. However, because of the thinness of the membranes and their internal variability, it has been hard to determine which aspects of the membranes most affect their performance. Culp *et al.* combined electron tomography, nanoscale three-dimensional (3D) polyamide density mapping, and modeling of bulk water permeability with zero adjustable parameters to quantify the effect of 3D nanoscale variations in polymer mass on water transport within the polyamide membrane (see the Perspective by Geise). They found that variability in local density most affects the performance of the membranes. Better synthesis methods could thus improve performance without affecting selectivity.

Science, this issue p. 72; see also p. 31

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