Section S1. Calculations of the limiting dimensions

HI nanofiltration membranes function by an ordered array of hexagonally close-packed nanofibrils with well-defined center-to-center spacings from \( d = 3.1 \) to 3.9-nm. The prior study suggests the swelling by water is negligible. Here because the polymeric nanostructures from lyotropic self-assembled mesophases were cross-linked and anchored by covalent bonds, we assume the influence of solvent swelling is negligible. The solvent continuous nanochannel schematic listing the nomenclature of the geometric parameters is shown in Figure S5 below. Note the dissociation of the counterion \( \text{Br}^- \) from \( \text{NH}_4\text{Br} \) depends on the solution dielectric constant, \( \varepsilon \). Hence, the dissociated \( \text{Br}^- \) may be presented as a part of the solvent continuum in aqueous solutions; alternatively, a larger diameter cylinder can be expected if there is no ionic dissociation. In turn, 2 cylinder volume fractions \( \varphi \) for each mesophase are approximated. The limiting dimension is derived from the below calculations:

\[
l = \frac{2d}{3} \quad (S.1)
\]

\[
\delta = l - r_f \quad (S.2)
\]

\[
t = l \cdot \sin\left(\frac{\pi}{3}\right) \quad (S.3)
\]

\[
2\delta = d \left[ \frac{4}{3} - \left( \frac{8\varphi}{\sqrt{3\pi}} \right)^{\frac{1}{2}} \right] \quad (S.4)
\]

\[
S_x = 2t - 2r_f = d \left[ \frac{4}{3} - \left( \frac{8\varphi}{\sqrt{3\pi}} \right)^{\frac{1}{2}} \right] \quad (S.5)
\]

\[
S_y = 3l - 2r_f = d \left[ 2 - \left( \frac{8\varphi}{\sqrt{3\pi}} \right)^{\frac{1}{2}} \right] \quad (S.6)
\]
Section S2. Calculations for the transport characterization

Hydraulic Permeance Calculation

For ordered cylinders with low Reynolds fluid flow perpendicular to the long axis, the dimensionless hydraulic permeability $k^*$ of can be approximated as a function of solid volume fraction $\varphi$ (45),

$$k^* = \frac{2\sqrt{2}}{9\varphi} (1 - \sqrt{\frac{4\varphi}{\pi}})^{5/2}$$  \hspace{1cm} (S.7)

Similarly, the dimensionless hydraulic permeability $k_{\parallel}^*$ of fluid flow parallel to the square array of fibers is (46):

$$k_{\parallel}^* = -\ln(\varphi) - 1.476 + 2\varphi - 0.5\varphi^2 - O(\varphi^4)$$  \hspace{1cm} (S.8)

These calculations lead to the Darcy permeability from the dimensionless permeability factor $k^*$ derived above.

$$k = k^* \cdot r_f^2$$  \hspace{1cm} (S.9)

From the above correlation, the hydraulic permeability $P$ is estimated by accounting for the water viscosity $\mu$ that is 0.0011 Pa·s at 16°C:

$$P = \frac{k}{\mu}$$  \hspace{1cm} (S.10)

Where the $P$ is directly related to the hydraulic permeance by factors of membrane thickness $\delta$ and tortuosity $\tau$,

$$L_p = \frac{P}{\delta}$$  \hspace{1cm} (S.11)

When there are mixed cylinder orientations (i.e., parallel cylinders followed with perpendicular cylinders) across the membrane cross-section, the associated hydraulic permeance $L_p$ is estimated with flow units in series,

$$\frac{1}{L_p} = \frac{1}{L_{p,1}} + \frac{1}{L_{p,2}} + \cdots + \frac{1}{L_{p,n}}$$  \hspace{1cm} (S.12)
The Calculation for Transport Characterization

The observed solute rejection $R_o$ was directly calculated by comparing the solute concentration of the permeate to the feed. In the equation below, $C_p$ is the permeate concentration, and $C_f$ is the feed concentration,

$$R_o(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (S.13)$$

In the aqueous solution rejection (PEG) experiment, the intrinsic solute rejection $R_a$ most accurately reflects the nanochannel sieving property. It was calculated by correlating the observed rejection of the effect of concentration polarization by an equation of the volumetric water flux $J_w$ to the mass transfer coefficient $k$. Since the $J_w/k$ are always smaller than 0.3, there was no severe concentration polarization in aqueous solution experiments (47).

$$R_a(\%) = 1 - R_o + R_o \exp\left(\frac{J_w}{k}\right) \quad (S.14)$$

The concentration polarization correction was between 0.3 to 3% through the entire PEG rejection experiment. The mass transfer coefficient is estimated based on the Colton-Smith empirical correlation defined by a function of projected stir bar radius $r$, solute diffusion coefficient $D_i$, solvent kinematic viscosity $v$, and the rotational speed $\omega$ of the magnetic stirring bar,

$$\frac{kr}{D_i} = 0.285 \cdot \left(\frac{v}{D_i}\right)^{\frac{1}{3}} \cdot \left(\frac{\omega r^2}{v}\right)^{0.567} \quad (S.15)$$

Here, the $D_i$ values for polyethylene oxide molecules are retrieved from the literature. For other solutes, the $D_i$ is approximated using the Stoke-Einstein equation,

$$D_i = \frac{k_BT}{6\pi r_s} \quad (S.16)$$

In order to describe the rejection profile of H$_4$ templated membranes, a logistic function is employed to fit the sigmoidal-like progression of the molecular weight cut-off curve. By corresponding the solute diameter estimated from the Chem3D software package to the observed rejection, the fitting parameters $\beta$ and $\gamma$ best describing the rejection profile were derived (56),

$$R_a(\%) = \left(\frac{1}{1 + e^{-\gamma d_s + \beta}}\right) \times 100\% \quad (S.17)$$
Supplementary Figures

Fig. S1. Polarized optical micrographs (POM) displaying the high-fidelity retention of the mesophase textures. Micrographs were taken before and after the photo-induced polymerization for n5t, n6t, n6d, and n7d in thin-film. 20 wt.% mesophase solutions were spin-coated on pre-cleaned glass slides to prepare ~500 nm thick films. Consistent liquid crystal textures were observed without the emergence of optical inhomogeneities in the developed birefringence domains. Gel samples were prepared from ~20 wt.% solutions.
Fig. S2. POM images showing the disruption of the original mesophase texture after the polymerization of n8d mesophase. The micrograph was captured from a thin-film cast on a pre-cleaned glass slide. The fading of birefringence texture in the cross-linked sample indicates potential phase transformation.

Fig. S3. Grazing-incidence small-angle X-ray scattering (GISAXS) data of the n8d thin-film prepared through the direct solution coating of the precursor solution on silicon substrates. The polymerized nanostructures were analyzed for thin-films (A) prepared on an unmodified substrate and (B) cast on poly(vinylpyrrolidone) modified surface. Subsequently, the 1D SAXS data was calculated from 2D images shown in (C). The X-ray data suggest the phase transformation emerges during the polymerization of n8d mesophase. The peak spacing ratio $1: \sqrt{3}: \sqrt{4}: 3: 4$ in the polymerized film evidenced the co-existence of the lamellar and hexagonal close-packed nanostructure. Meanwhile, for the thin-film polymerized on PVP modified surface, the spacing ratio $1: 2: 3: 4$ characteristic for the lamellar nanostructure is observed.
Fig. S4. GISAXS patterns of the cross-linked n5t mesophases initiated from different surfactant concentrations. Mesophases were cast with concentrations as indicated (81 to 56 wt.%) atop of PVP modified substrate. The concentration of the cross-linker is kept at 4wt%, but the glycerol content was adjusted accordingly. The anisotropic pattern is mostly indiscernible from different mesophases examined.
Fig. S5. Schematic for the critical dimension of the hexagonally packed nanofibrils available for nanofiltration. Data are tabulated. The geometrical spacing calculations are estimated in solvent environments with different dielectric constants. The bromide ion is assumed to dissociate from the fibril matrix in solvent with a high dielectric constant or bind with the matrix in solvent with a low dielectric constant, and hence the different volume fractions. The grey particle represents the limiting dimension available for solvent flow along the axial direction, and the green particle is the confining dimension in case of the solvent flow perpendicular to the cylinders.
Fig. S6. GISAXS data and transport limiting dimension analysis for additional mesophases. The thin-films were prepared through spin-coating the mesophase solution atop the silicon substrates, followed by photoinitiated polymerization. (A) Tabulated mesophase formula prepared for the structural characterization. (B) 2D GISAXS images of polymerized mesophases indicate the H₁ was retained, and the cylinders remained in-plane morphology. (C) 1D SAXS data demonstrate the tunability in the center-to-center distance defined by H₁ mesophase compositions. A consistent spacing ratio 1: √3: √4 is followed. (D) Tabulated data of the critical dimensions. The sₓ column represents the transport limiting dimension assuming an in-plane cylinder orientation.

| Mesophase | Surfactant (% by weight) | X-linker (% by weight) | X-linker type | Glycerol (% by weight) |
|-----------|--------------------------|------------------------|---------------|------------------------|
| n6d-81    | 81                       | 4                      | HDDA          | 15                     |
| n7d-72    | 72                       | 3                      | HDDA          | 25                     |
| n7t-55    | 55                       | 3                      | PETA          | 42                     |
| n7t-72    | 72                       | 3                      | PETA          | 25                     |

| Mesophase | Cylinder fraction (% by volume) | d-spacing (nm) | 2r₁ (nm) | sₓ (nm) | 2δ (nm) |
|-----------|---------------------------------|---------------|----------|---------|---------|
| n6d-81    | 67.6                            | 3.2           | 3.19     | 0.566   | 1.08    |
| n6t-76    | 64.4                            | 3.1           | 3.02     | 0.563   | 1.12    |
| n7t-72    | 61.5                            | 3.0           | 2.85     | 0.611   | 1.15    |
| n7t-55    | 61.1                            | 3.1           | 2.94     | 0.641   | 1.20    |
| n7t-72    | 53.3                            | 3.2           | 2.83     | 0.862   | 1.43    |
| n6t-61    | 52.9                            | 3.4           | 3.00     | 0.926   | 1.53    |
| n7t-55    | 48.7                            | 3.3           | 2.79     | 1.02    | 1.61    |
| n7t-55    | 48.4                            | 3.7           | 3.12     | 1.15    | 1.81    |
Fig. S7. High magnification POM images from additional prepared mesophases. Thin films were prepared from spin-coating the solution on pre-cleaned glass slides, followed by gentle heating to develop the birefringence domains. Micrographs were recorded before and after the photoinitiated cross-linking. Albeit the difference in compositions from their original formulas, consistent focal conic textures were identified before and after the polymerization.
Fig. S8. 1D GISAXS data of the n5t mesophases with different surfactant concentrations and the associated limiting dimension analysis. Mesophases were cast on PVP modified substrates. (A) Magnified 1D integrated data of the n5t with a consistent cross-linker concentration, but small increments in weight fractions from 56 to 81 wt.%. (B) The change in $d_{100}$ possesses a non-linear relationship to weight fractions. The non-monotonic correlation may have resulted from the interplay between cross-linker swelling and surfactant packing in the lyotropic phase. (C) A survey of limiting dimensions reveals the confined limiting dimensions are linearly related to surfactant weight fractions with precise increments ~0.1 nm. The limiting dimension in the radial direction is linear in the d-spacing and varies as the square root of the volume fraction. Thus, at higher weight fractions, the modest increase in the d-spacing ~3% is offset by the larger decrease in the square root of the volume fraction term $(4/3)^{1/2} - (8\phi/\sqrt{3}\pi)^{1/2} \sim 40\%$. As a result, the transport limiting dimension continues to decrease with volume fraction, despite the increase in the d-spacing.
Fig. S9. Magnified micrographs of the thin-film composite membranes. The color-overlay represents the film thickness of the self-assembled thin membrane atop the PVDF ultrafiltration support.
Fig. S10. Estimating hydraulic permeance from different nanostructures from theory. (A) Here n6t, n6d, and n7d are modeled with a parallel orientation, and the n5t is considered to possess mixed cylinder orientations mostly in perpendicular cylinders. For n5t, parallel cylinders are assumed to project half of the surface area, and span across half of the film thickness. (B) The thickness normalized hydraulic permeabilities were calculated using the results from the structural analysis, plus assuming a consistent tortuosity ~2.5 for each membrane. (C) The calculated hydraulic permeances are similar to the measured permeances.
Fig. S11. A survey of 2D GISAXS patterns for the cross-linked n6d mesophases immersed in different organic solvents for a pre-determined amount of time. After the prolonged soaking period, films were dipped on towels and dried in high-vacuum prior to imaging. A consistent $d_{100}$ spacing ~3.4-nm was observed based on the 1D-integrated data, and there was no significant nanostructural disruption during this process.
Fig. S12. Solvent flux as a function of transmembrane pressure for different H₃ membranes. Each membrane is listed with four solvents, including water (labeled as red diamond), methanol (labeled as orange triangle), ethanol (labeled as blue triangle) and isopropanol (labeled as green square). The solvent permeance is derived from the linear regression as represented by the dashed line.
Fig. S13. The product of solvent permeance and viscosity normalized using the water value. The product is listed as a function of (A) the polarity factor in the Hansen solubility parameter and (B) the total Hansen solubility parameter for different H₁ membranes. A linear correlation is established. (C) A recreation of the Buekenhoudt flux model suggests the water flux normalized proportionality factor is linear with pore size. The calculations were based on the Hansen solubility parameters and the physical properties of the organic solvents tabulated in (D), data retrieved from literature (13, 48, 57).
0.6 kg mol\(^{-1}\) PEG molecule (in water):

Small molecules (in methanol or isopropanol):

Neutral Red (NR) 289 g mol\(^{-1}\)
Methylene Blue (MB) 320 g mol\(^{-1}\)
Crystal Violet (CV) 408 g mol\(^{-1}\)
Victoria Blue B (VB) 506 g mol\(^{-1}\)
Coomassie Brilliant Blue R-250 (CB) 826 g mol\(^{-1}\)

Methyl Orange (MO) 327 g mol\(^{-1}\)
Rose Bengal (RB) 973 g mol\(^{-1}\)
Acid Fuchsin (AF) 585 g mol\(^{-1}\)
Alcian Blue (AB) 1299 g mol\(^{-1}\)

Fig. S14. Schematic of spacing filling models for solutes involved in the solute separation experiments with arrows linking to the corresponding solute diameter. The geometric mean diameters are derived from the molecular dimensions estimated by the Chem3D software package for dye molecules, or directly retrieved from literature for PEG600.

Fig. S15. Methanol permeability and methyl orange rejection performance of the H\(_{1}\) membranes. Membrane performance is listed along with literature data. Here the self-assembled membranes stand out with superior solvent permeability relative to amorphous materials at high solute rejections. Error bars correspond to 95% confidence interval from multiple measurements.
Fig. S16. UV-visible (UV-vis) spectra and photographs of the permeate collected from isopropyl alcohol (IPA) solution. The data are showing the solute separation of acid fuchsin (AF) and methyl orange (MO) permeate from the $H_i$ membranes in IPA. Note the n7d membrane, the $H_i$ with the largest theoretical pore size almost completely rejected AF ($\sim 590$ g mol$^{-1}$). Photo credit: Yizhou Zhang, University of Pennsylvania.
Fig. S17. Transmission electron micrograph for the CdSe nanoparticle reveals a spherical solute geometry with a diameter of 2.7-nm.

Fig. S18. UV-vis spectra and photographs demonstrating the complete solute separation of the CdSe nanoparticle dissolved in IPA by n6d and n7d membranes. Photo credit: Yizhou Zhang, University of Pennsylvania.
Fig. S19. UV-vis spectra and photographs showing the competitive solute separation of the CdSe nanoparticle and methylene blue (MB) dissolved in IPA by n6d and n7d H1 membranes. Photo credit: Yizhou Zhang, University of Pennsylvania.

Fig. S20. An enhancement in the photoluminescence intensity is observed after the rejection of CdSe nanoparticle dispersed in an equal volume of IPA and hexane by a n6d membrane. The solution was normalized to the same nanoparticle concentration. While the nanoparticle is not fluorescent in IPA solution, the enhancement may evidence a potential solvent exchange behavior with enriched hexane in the feed.
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