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Supporting information for article:

Innovative pressure environment combining hydrostatic pressure gradient and mechanical compression for structural investigations of nanoporous soft films

Julie Wolanin, Jérôme Giraud, Isabelle Morfin, Anne-Laure Rollet, Laurent Michot and Marie Plazanet

S1. Cell drawings

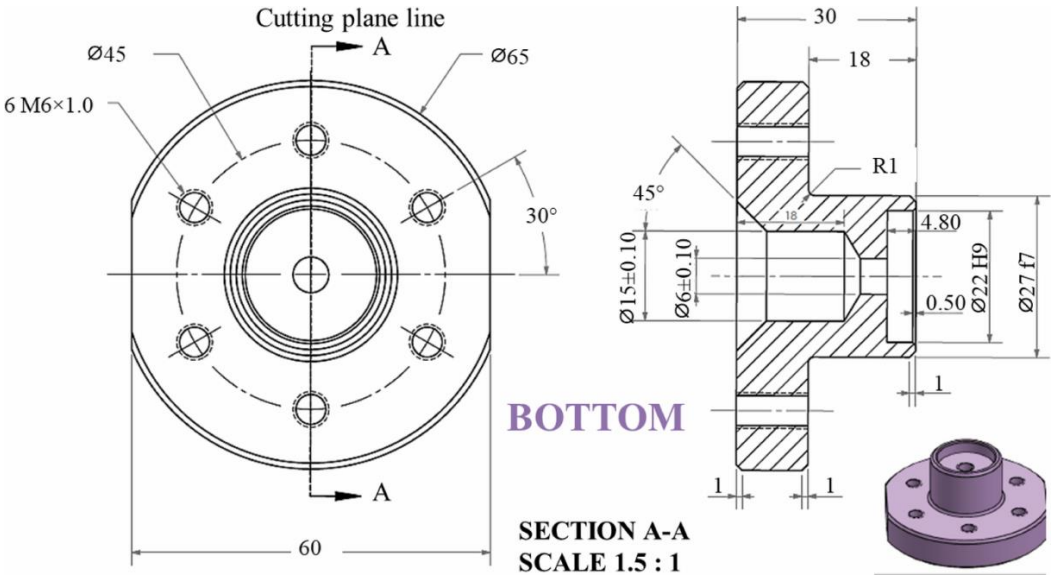

Figure S1 Drawings of the top and bottom parts of the cell - bottom.

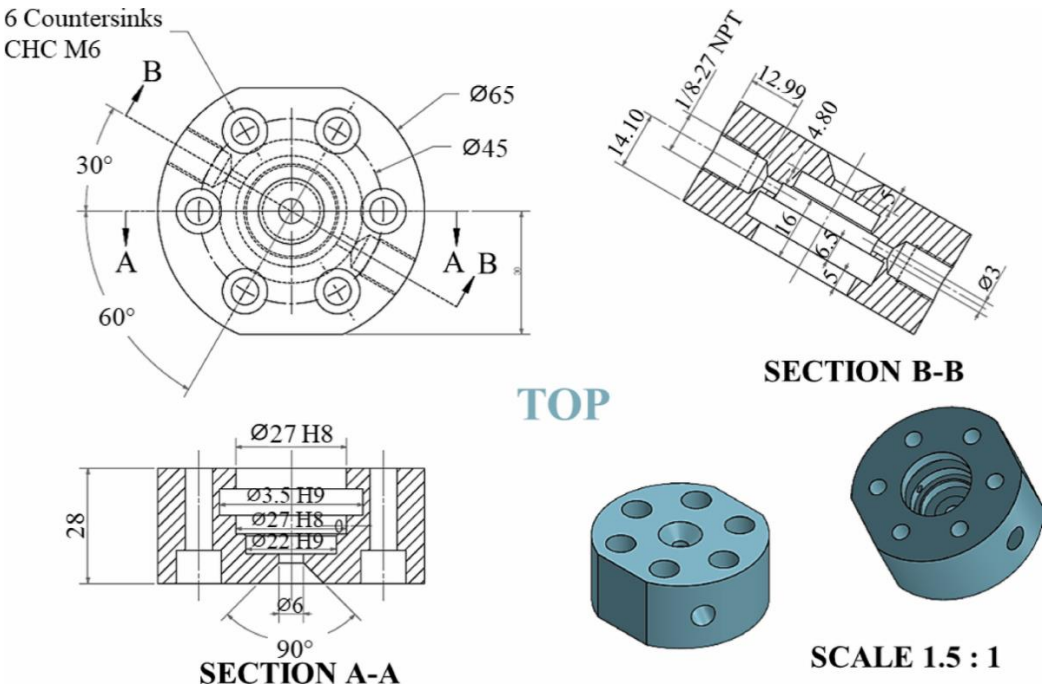

Figure S2 Drawings of the top and bottom parts of the cell - top.

S2. SAXS measurement on regenerated cellulose membrane

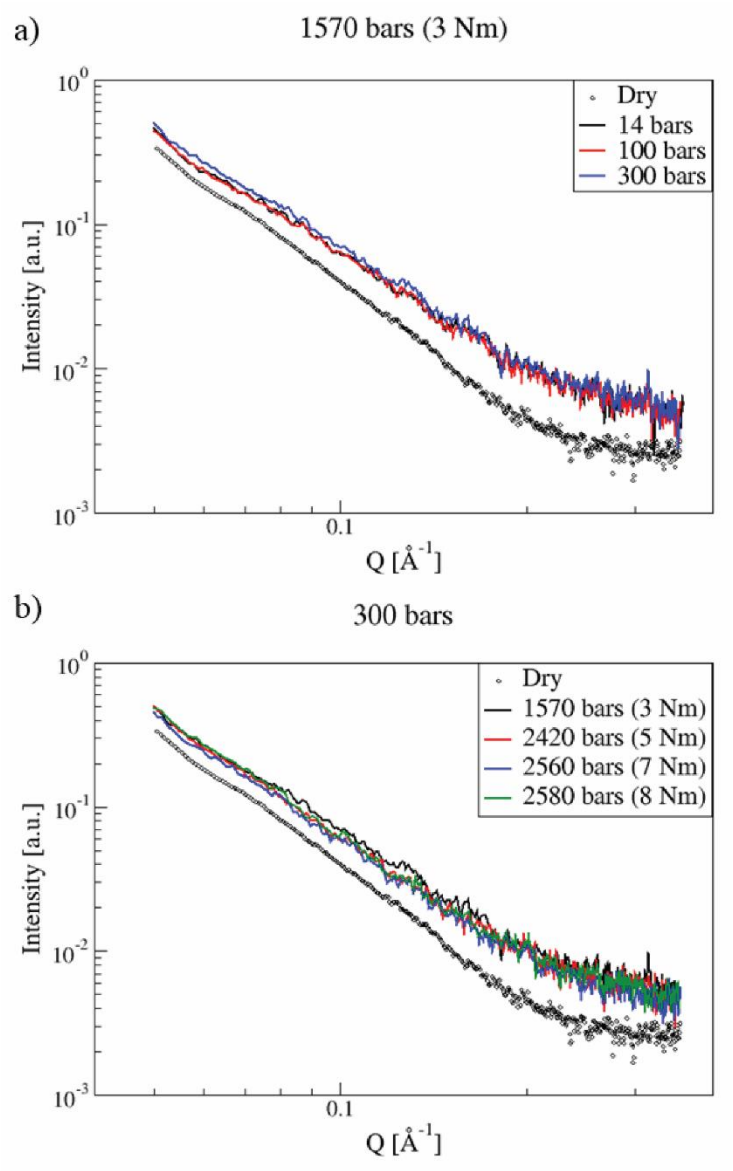

Figure S3 Small Angle signal measured on a cellulose.

S3. Example of SAXS data measured on Nafion membrane

Similar measurements have been performed with a perfluorosulfonated membrane (Nafion type). Briefly, perfluorinated sulfonic acid membranes consists of hydrophobic (Teflon type) backbone and hydrophilic side chains terminated by sulfonic acid groups. In the analyzable SAXS Q-range with our pressure cell, i.e. for Q higher than 0.05 $Å^{-1}$, the ionomer peak is observed. This well-defined maxima has already been characterized in the literature and is attributed to nanophase separation: its position is associated to the characteristic distance between the polymeric aggregates (Kusoglu & Weber, 2017). Rubatat et al. (Rubatat et al., 2002) have demonstrated the formation of elongated polymeric aggregates (cylindrical or ribbon-like aggregates). This ionomer peak is known to shifts continuously toward small-angle as the water content is increased.

The presented data have been obtained with three overlaid Nafion 112 membranes (purchased from du Pont de Nemours). Before measurements, the membranes were pre-treated repeating twice the following protocol: they were soaked for 2h in 1M HCl, then for 2h in 1 M NaOH. Finally, the membranes were boiled for 1h in deionized water and dried. The following figures show the evolution of the ionomer peak as a function of hydrostatic (14, 100 and 300 bars) or compressive (3, 5 and 8 Nm) pressures.

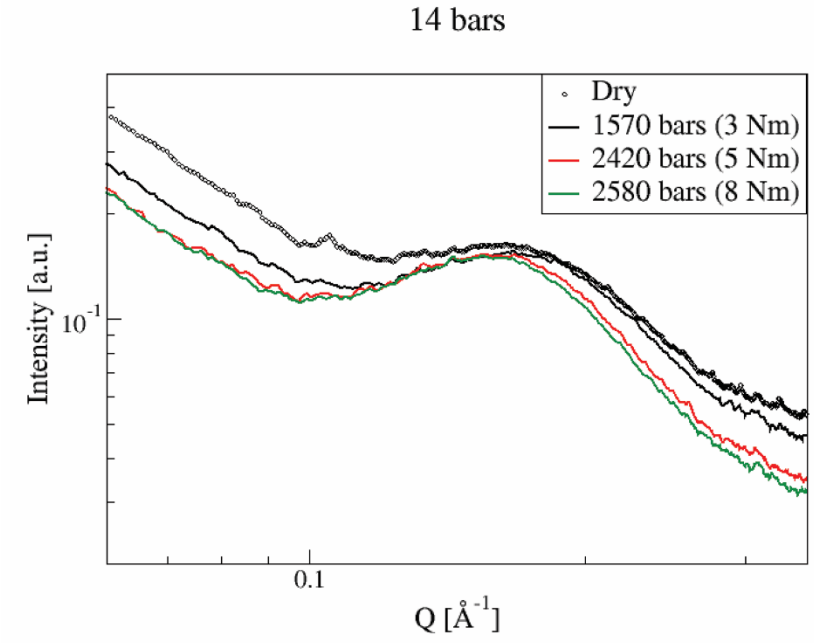

Figure S4 Small Angle signal measured on a Nafion membrane.

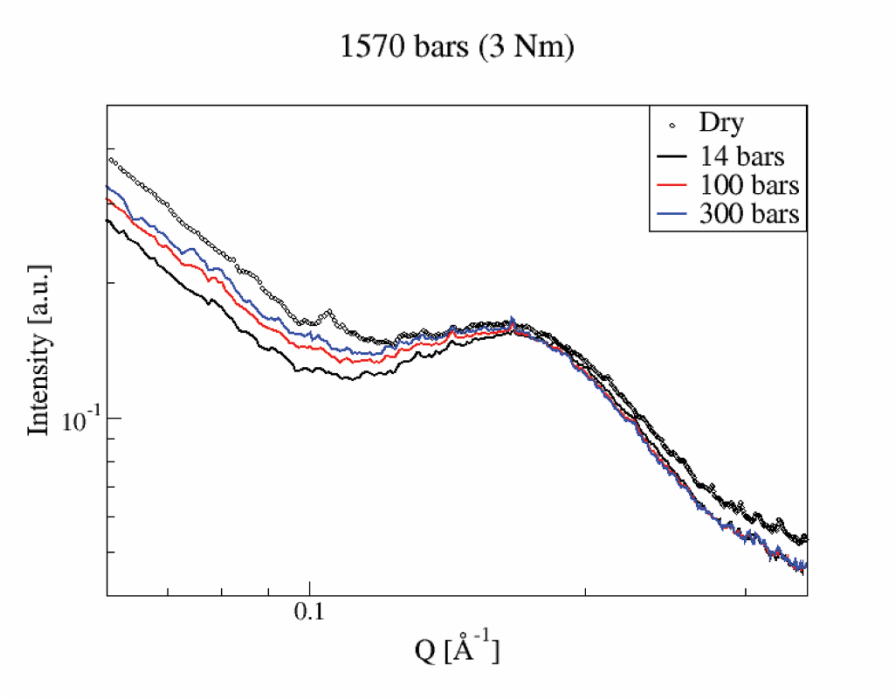

Figure S5 Small Angle signal measured on a Nafion membrane.

References

Kusoglu, A. & Weber, A. Z. (2017). *Chemical Reviews*, **117**(3), 987–1104.

Rubatat, L., Rollet, A., Gebel, G. & Diat, O. (2002). *Macromolecules*, **35**(10), 4050–4055.