Condensation and Evaporation of Hexane in Nanoporous Alumina Membranes

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Overview

1. Context
2. Goals of the internship
3. Theoretical background
4. Experimental setup
5. Conclusions

Context

Grand scheme

- Condensation and evaporation of fluids in confinement

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- Dependency on
  - pore diameter
  - temperature (relative to the critical temperature)

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Plan

- Anodized alumina membranes (AAM)
- Test setup using Hexane $\rightarrow$ working at room temperature permits much faster executable experiments
- Transfer to **helium** experiment

Goals

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- Performing isotherm measurements on many membranes for **statistics**

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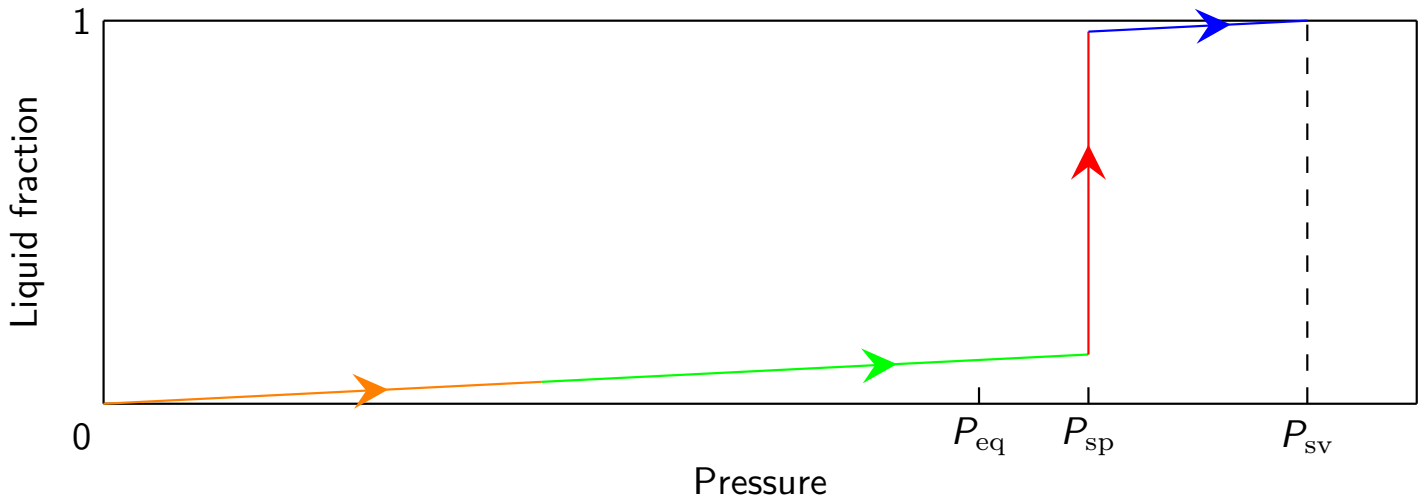
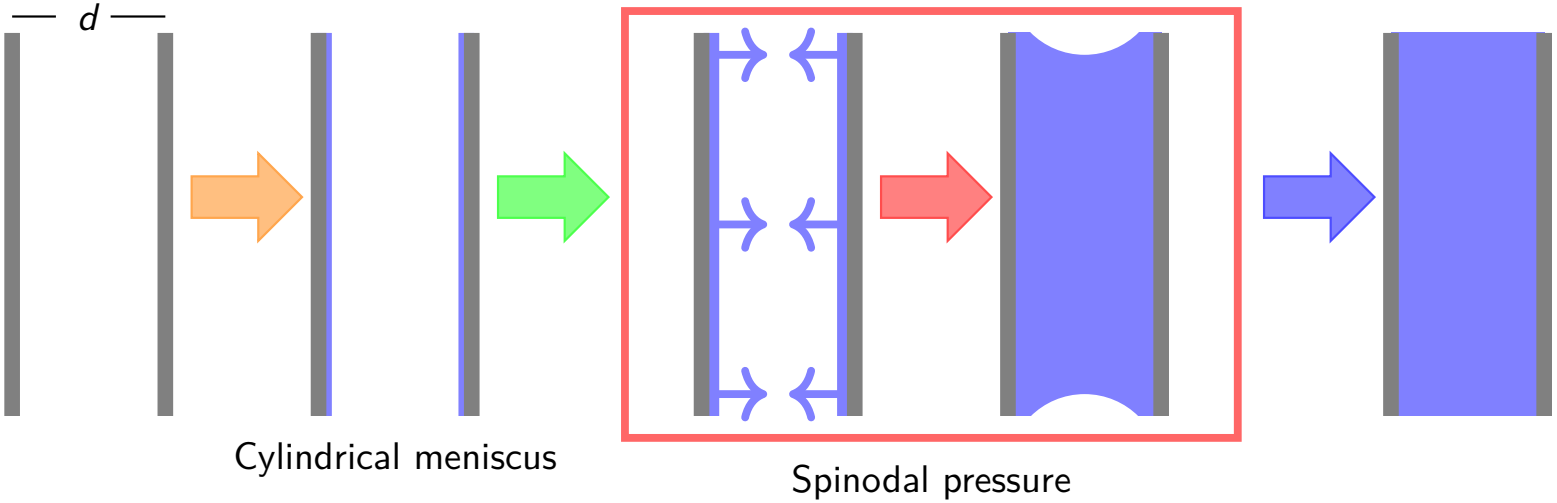
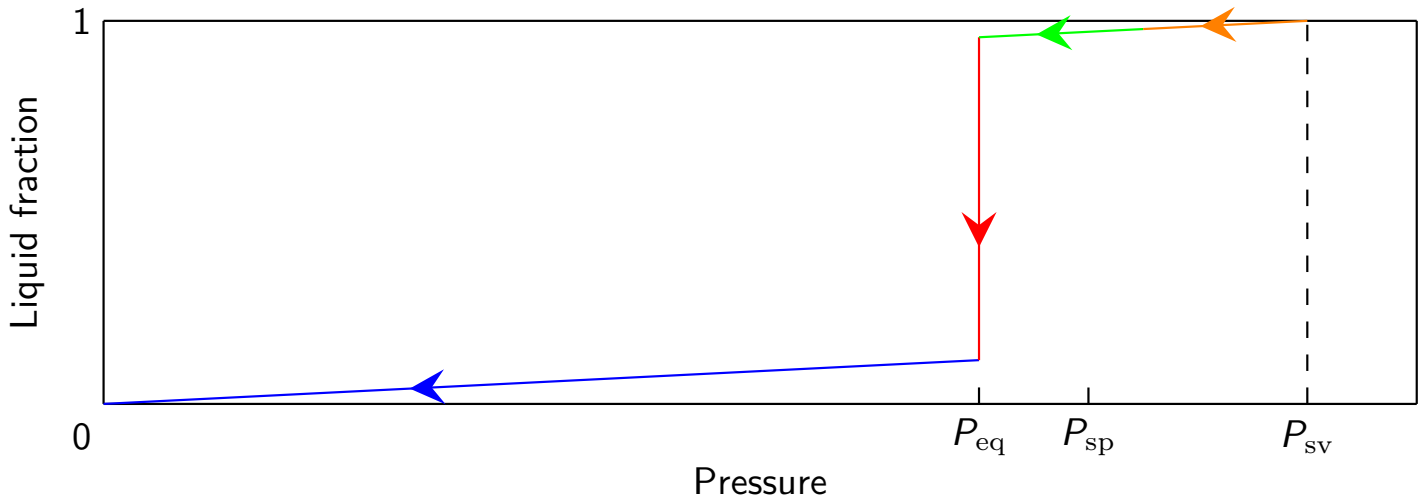
- Performing isotherm measurements on many membranes for **statistics**
- Comparing the pore diameters extracted from the volumetric measurements those from scanning electron microscopy (SEM) images
- Improving the fabrication process to reduce the dispersion
- Testing the efficiency of the ALD process as a means to reduce the pore diameters

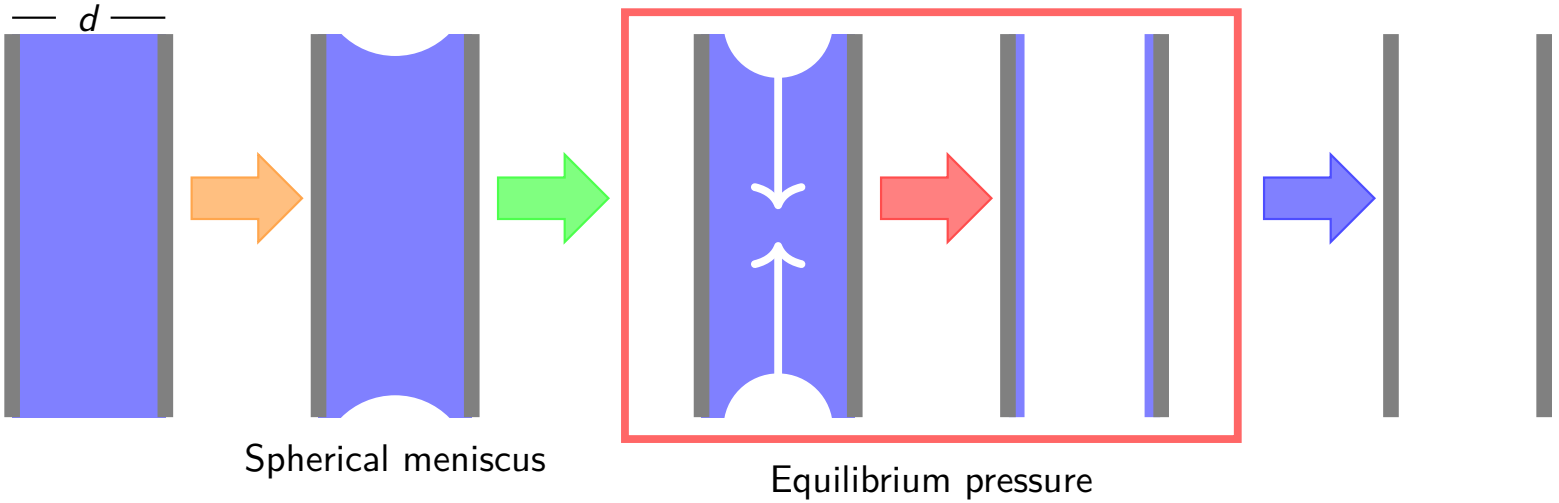
Condensation in a cylindrical open pore



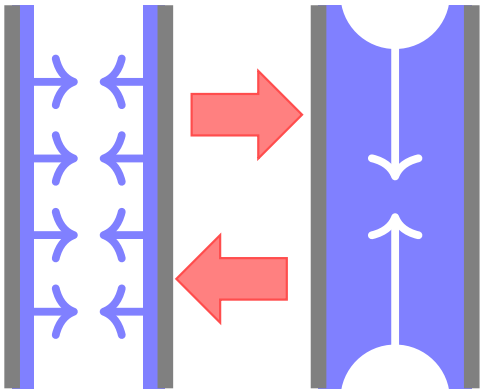


Evaporation in a cylindrical open pore



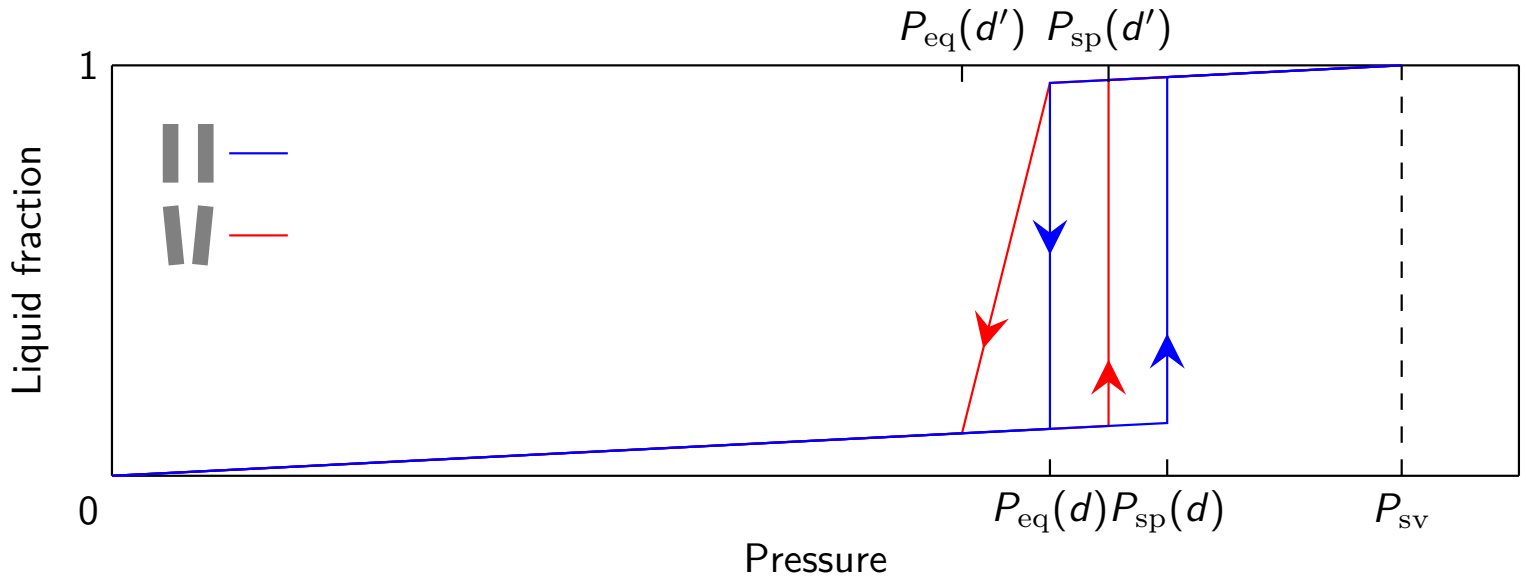


Condensation and evaporation in a cylindrical open pore

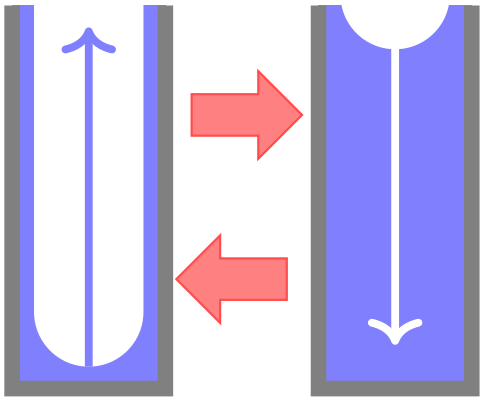


Open cylindrical pore

Condensation at spinodal pressure and evaporation at equilibrium pressure yield a **hysteresis.**

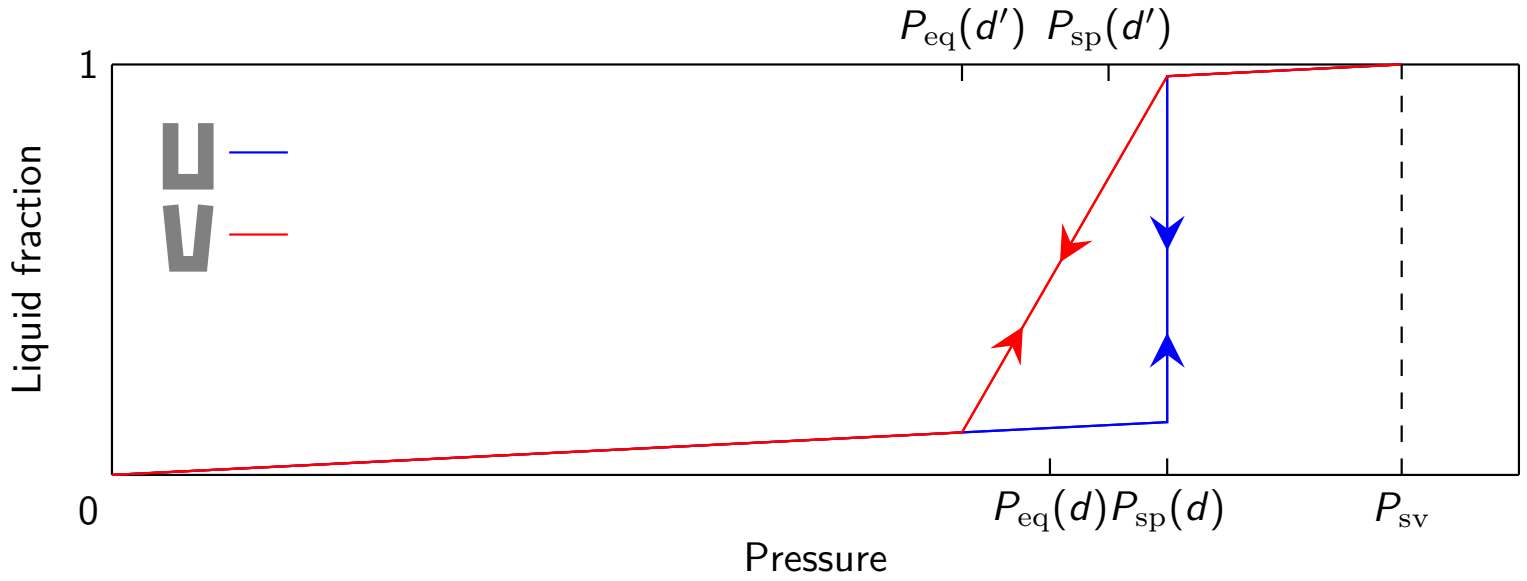


Condensation and evaporation in a cylindrical open pore



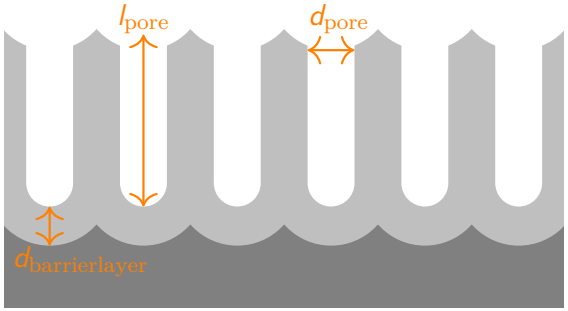
Closed cylindrical pore

Condensation at equilibrium pressure and evaporation at equilibrium pressure leads to **disappearance of the hysteresis.**

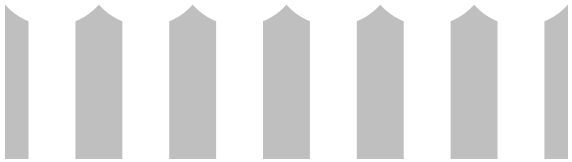


Membrane production

Two step anodizing
in oxalic acid
($C_2H_2O_4$) at 0 °C

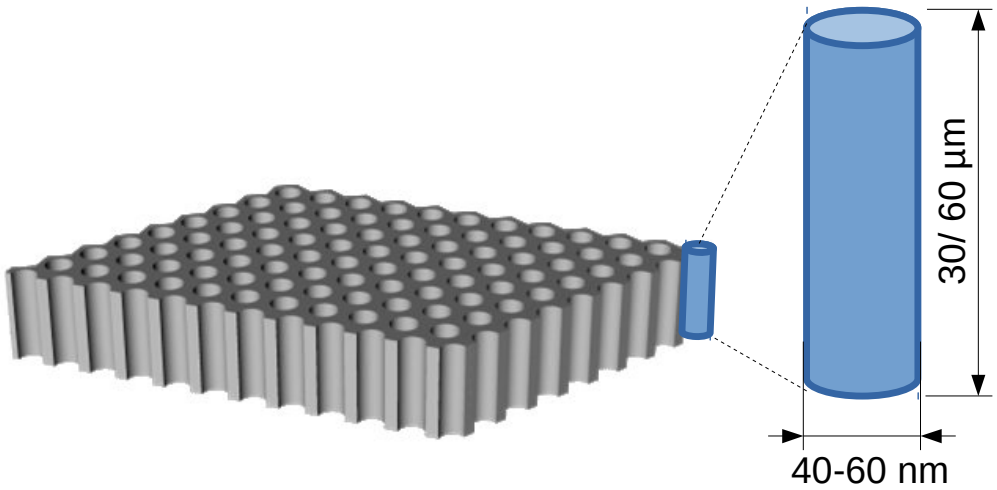


Immersion in
$\underbrace{27.2\,g \quad CuCl_2}_{\text{copper chloride}}$
$+\underbrace{0.2\,l \quad HCL(37\,\%)}_{\text{hydrochloric acid}}$
$+\underbrace{0.8\,l \quad H_2O}_{\text{water}}$



Floating on
phosphoric acid
(H_3O_4P) till milky
aspects appear
plus 15 min

Alumina membranes - funnellization



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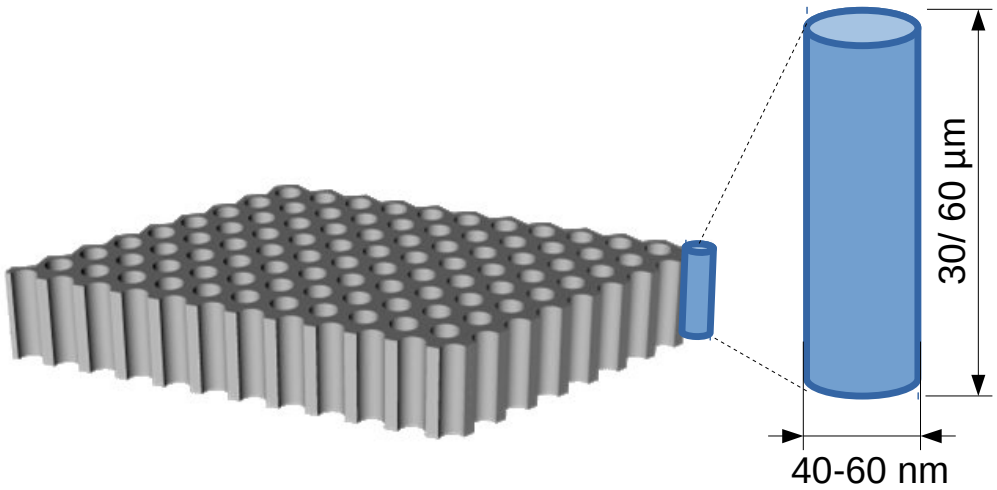

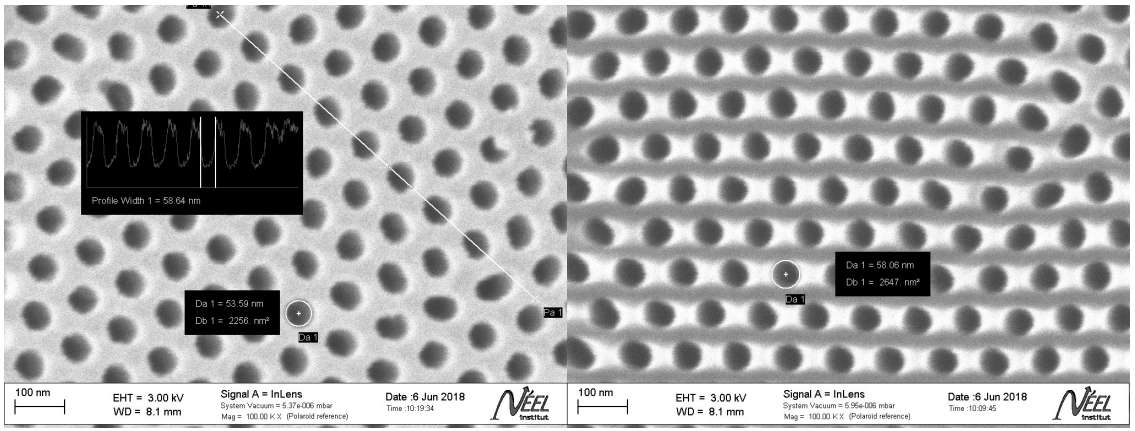

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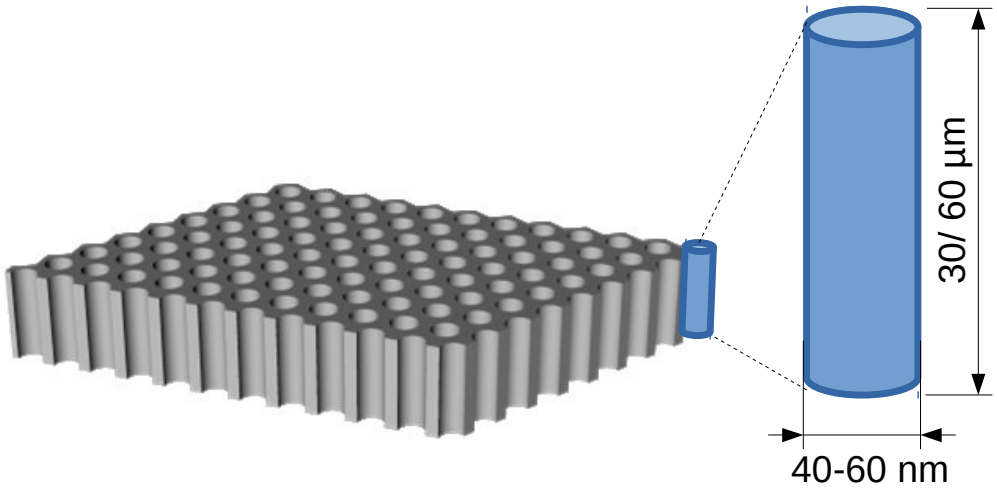
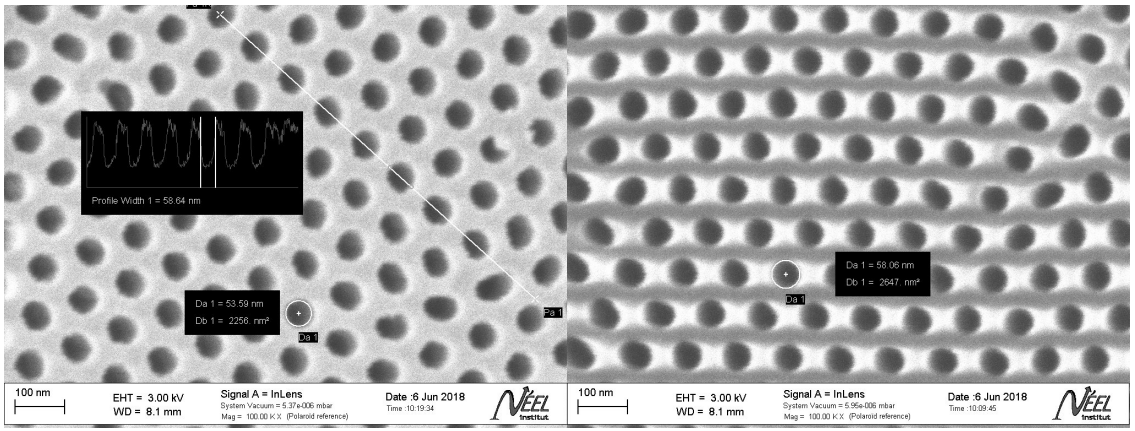
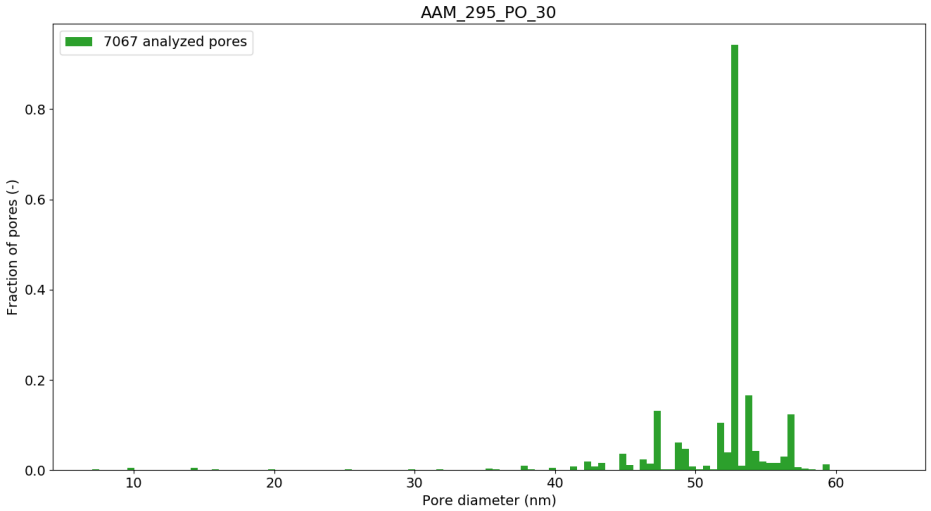


Funnellization

Funnellization suspected due to different pore diameters on top and bottom side.



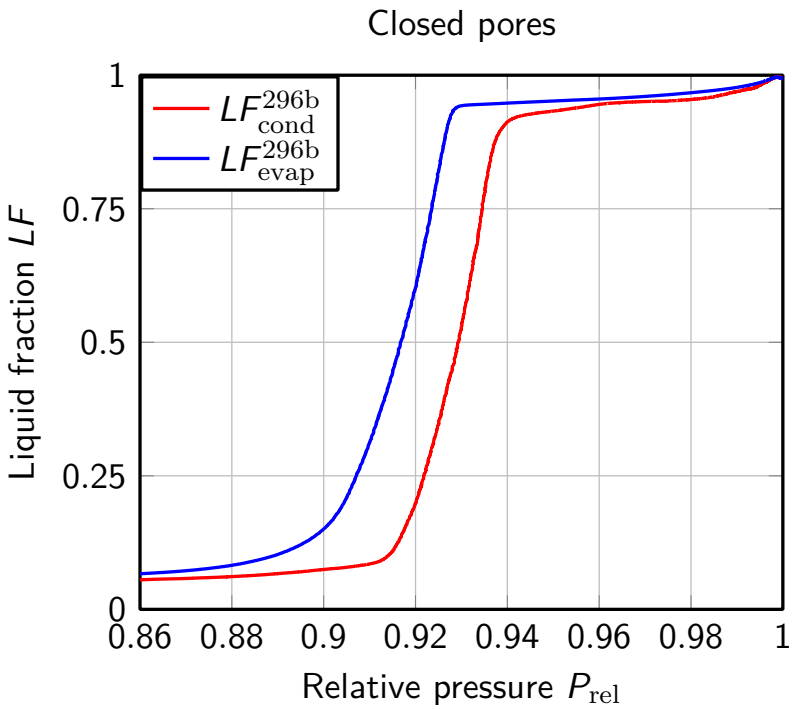
Alumina membranes - pore size distribution



Pore size distribution

SEM analysis shows pore size distribution on a given membrane.

Alumina membranes - corrugations



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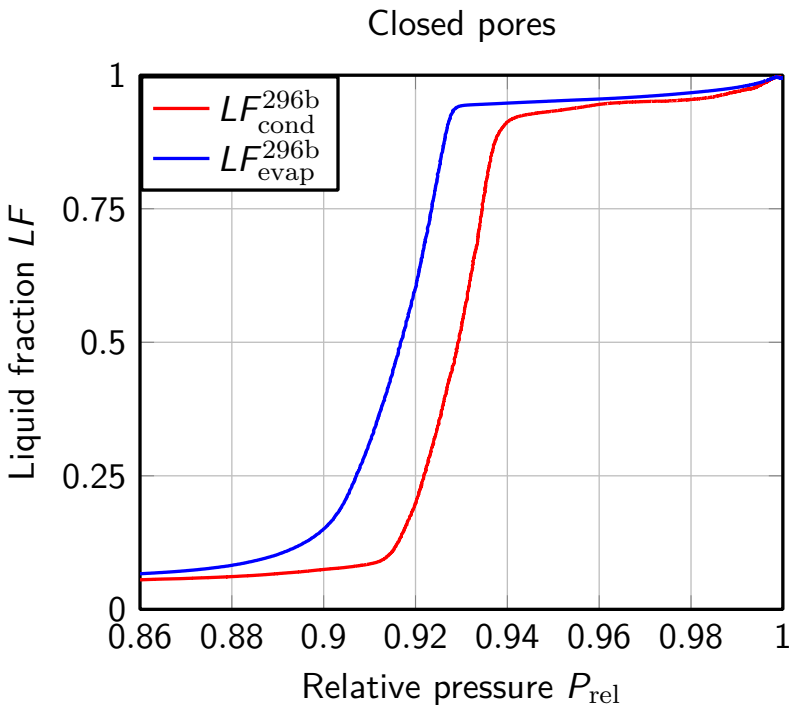
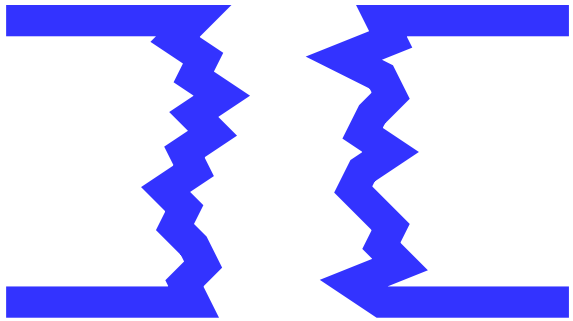


Corrugations

The appearance of the hysteresis is assumed to be due to **intra pore corrugations**.



Alumina membrane defects

Isotherms are affected by

- Pore size distribution
- Funnellization
- Corrugations

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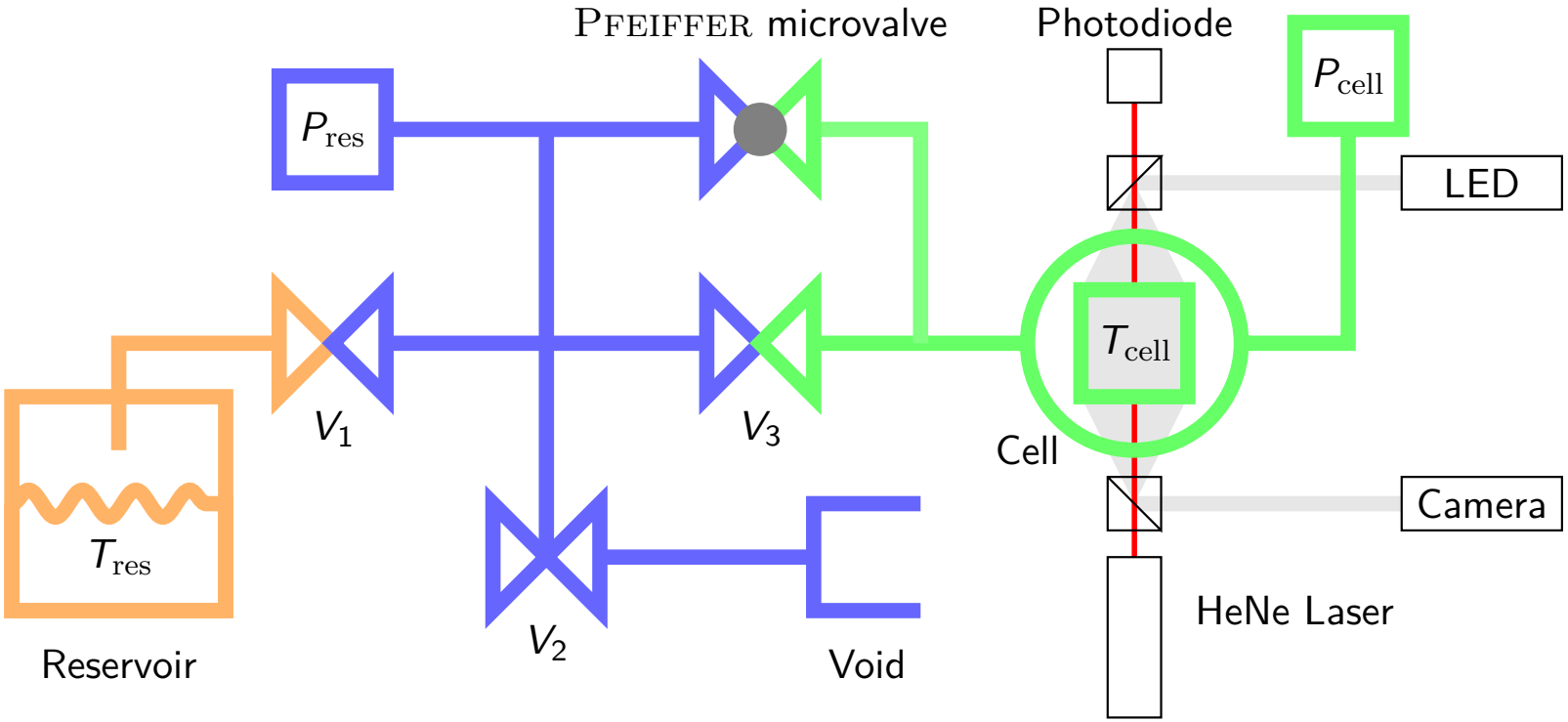
Problem

No simple ways to characterize these defects!
SEM images only give an impression of the surfaces of the membrane (factor between pore diameter and pore length is 1000!).

$\rightarrow$ Need of **monodisperse membranes**

Experimental setup



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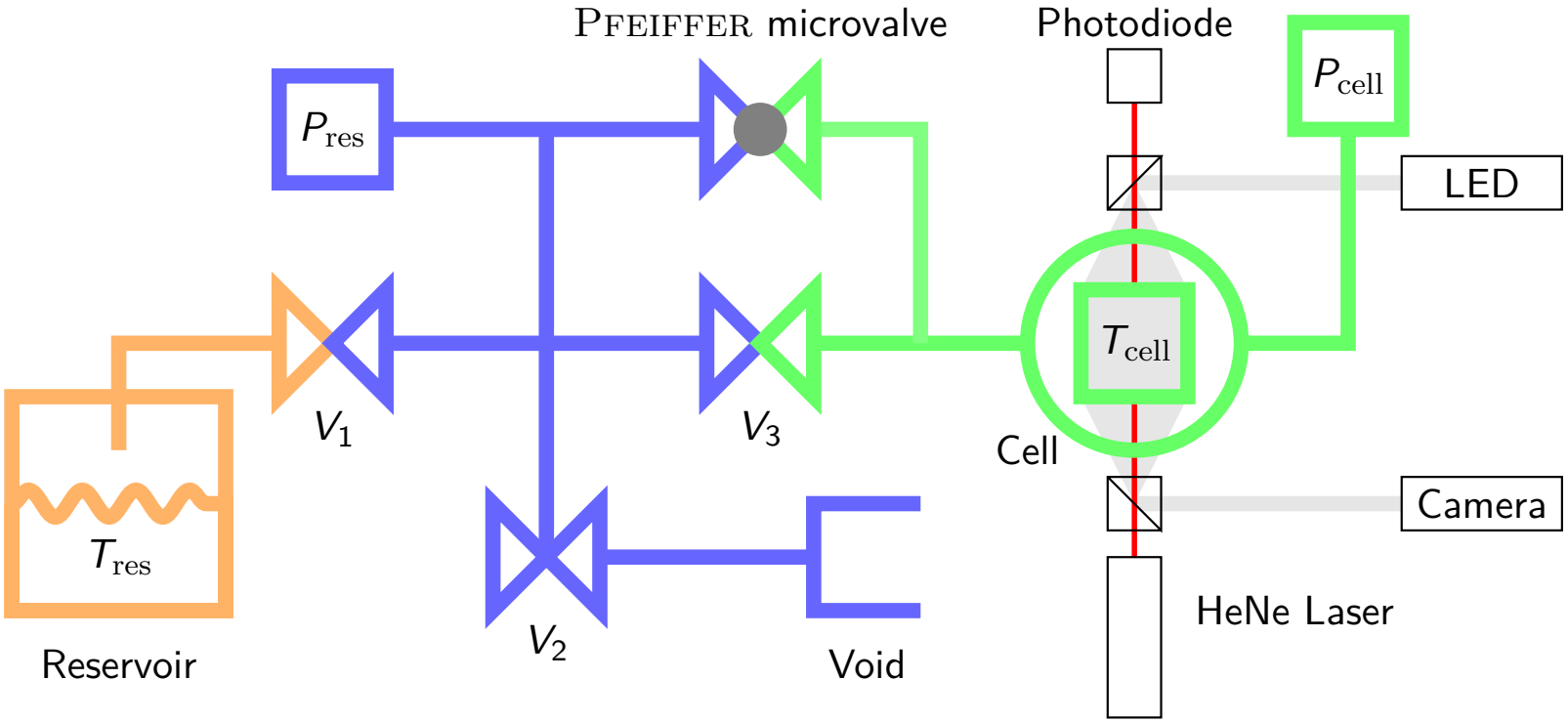

Volumetric and optical measurements

Volumetric and optical setups work independently.

Data evaluation

Inverse funnelling

Atomic layer deposition

SEM image analysis

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