

Preparation and Characterization of Alumina-Silica Composite Membranes for Modification by Atomic Layer Deposition Technique

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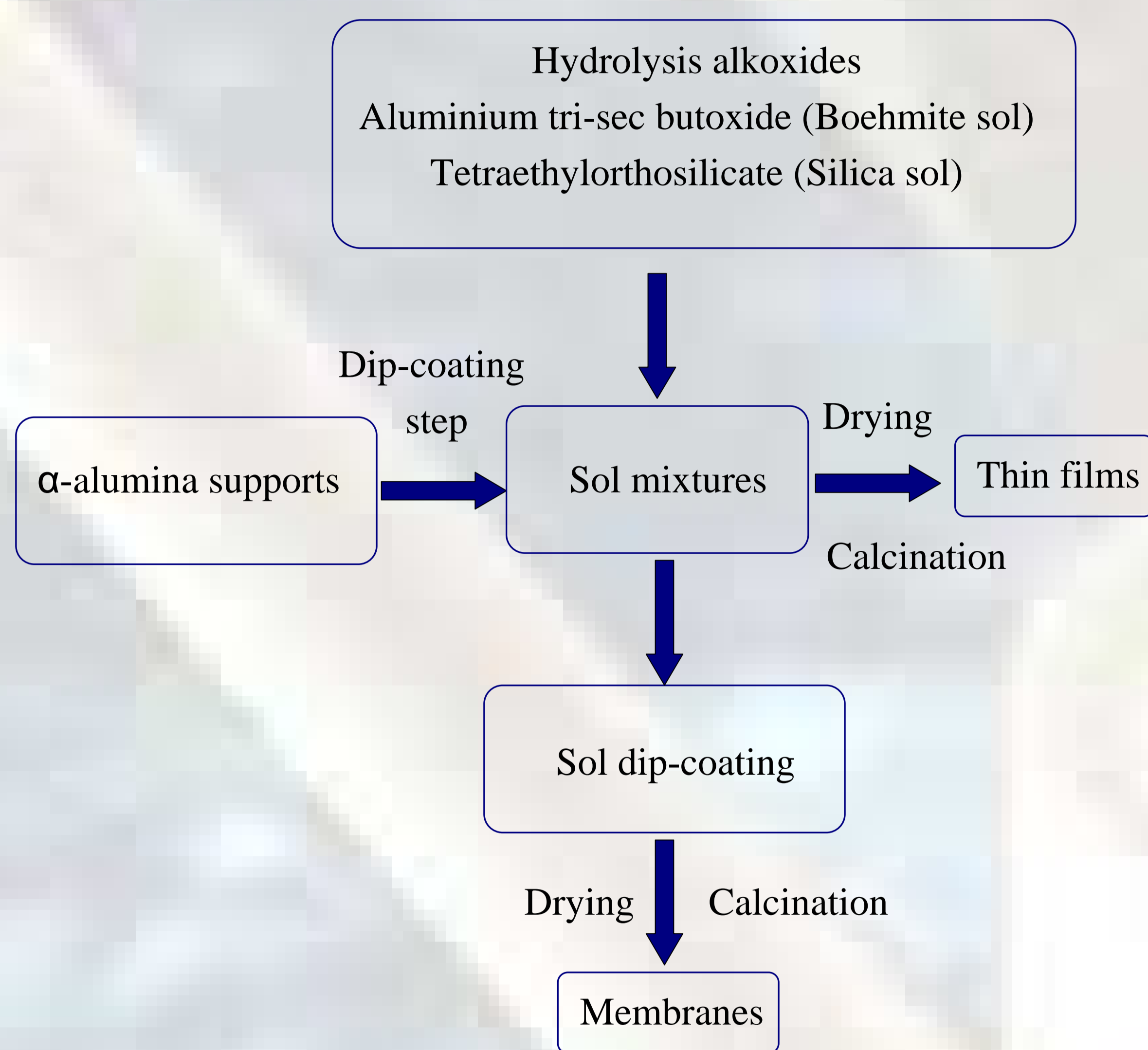
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Aim of research

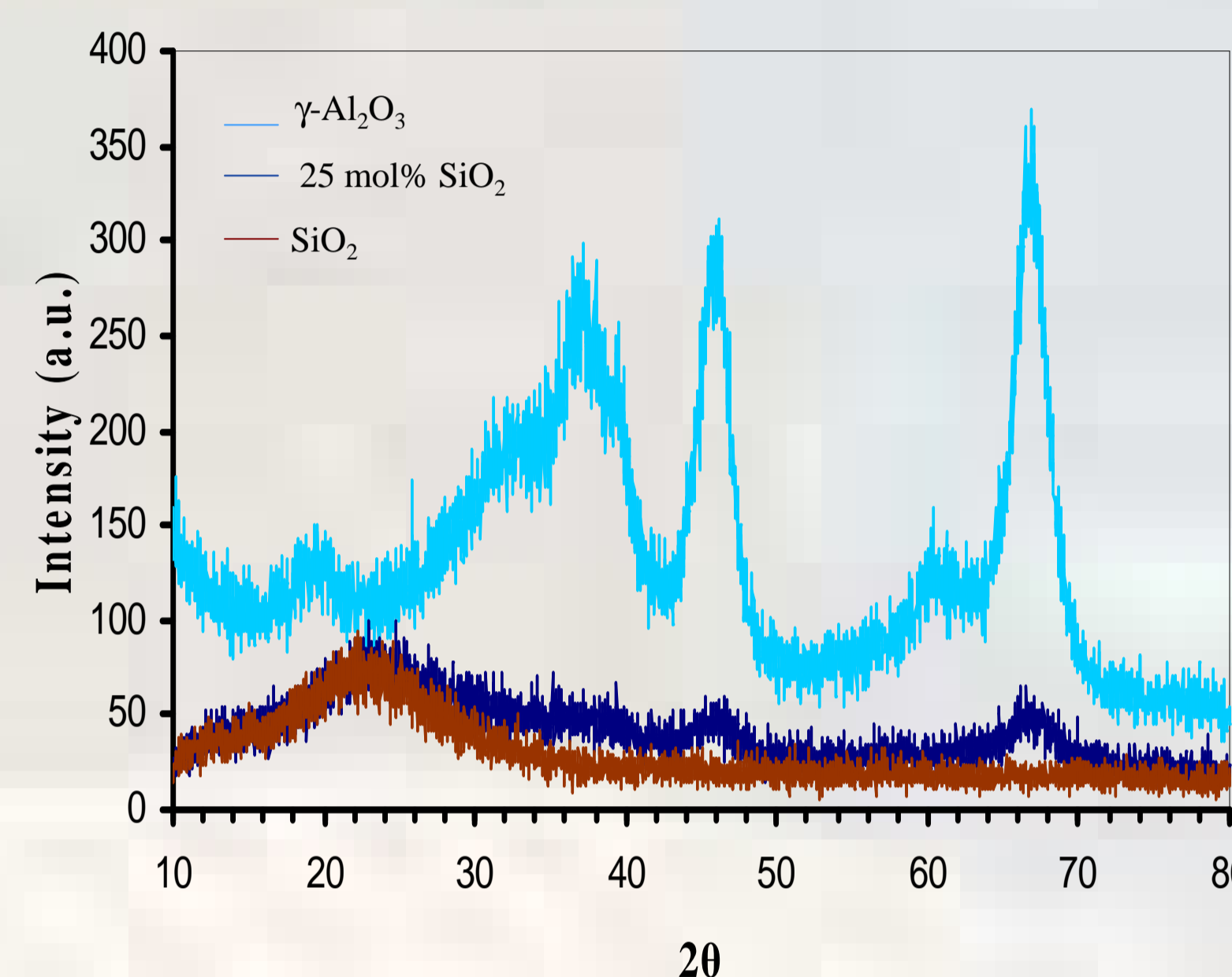
The synthesis and the characterization of membrane materials for hydrogen separation that will be used for the development of a membrane reactor for carbon free fossil fuel conversion is the main task of this research. Substrates made via the sol-gel technique are going to be modified by Atomic Layer Deposition in order to tune the size of the pores and protect against hydrothermal attack. A narrow pore size distribution for the starting materials is a prerequisite for an excellent separation performance because the tolerance for the sieving effect is small for a H₂-CO or H₂-CO₂ mixture.

Experimental

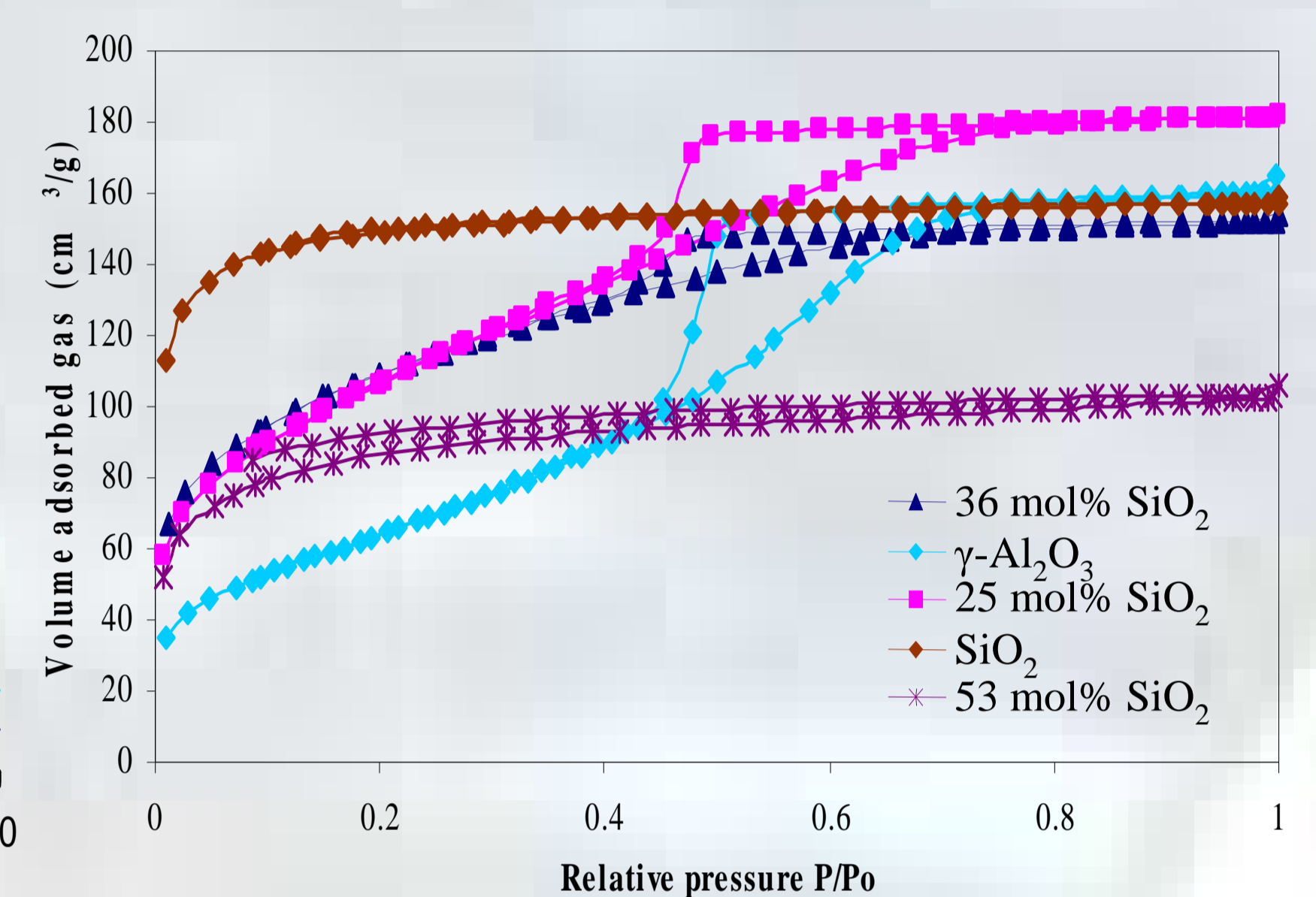
Sol- Gel steps for the preparation of Al₂O₃ - SiO₂ membrane materials



Characterization

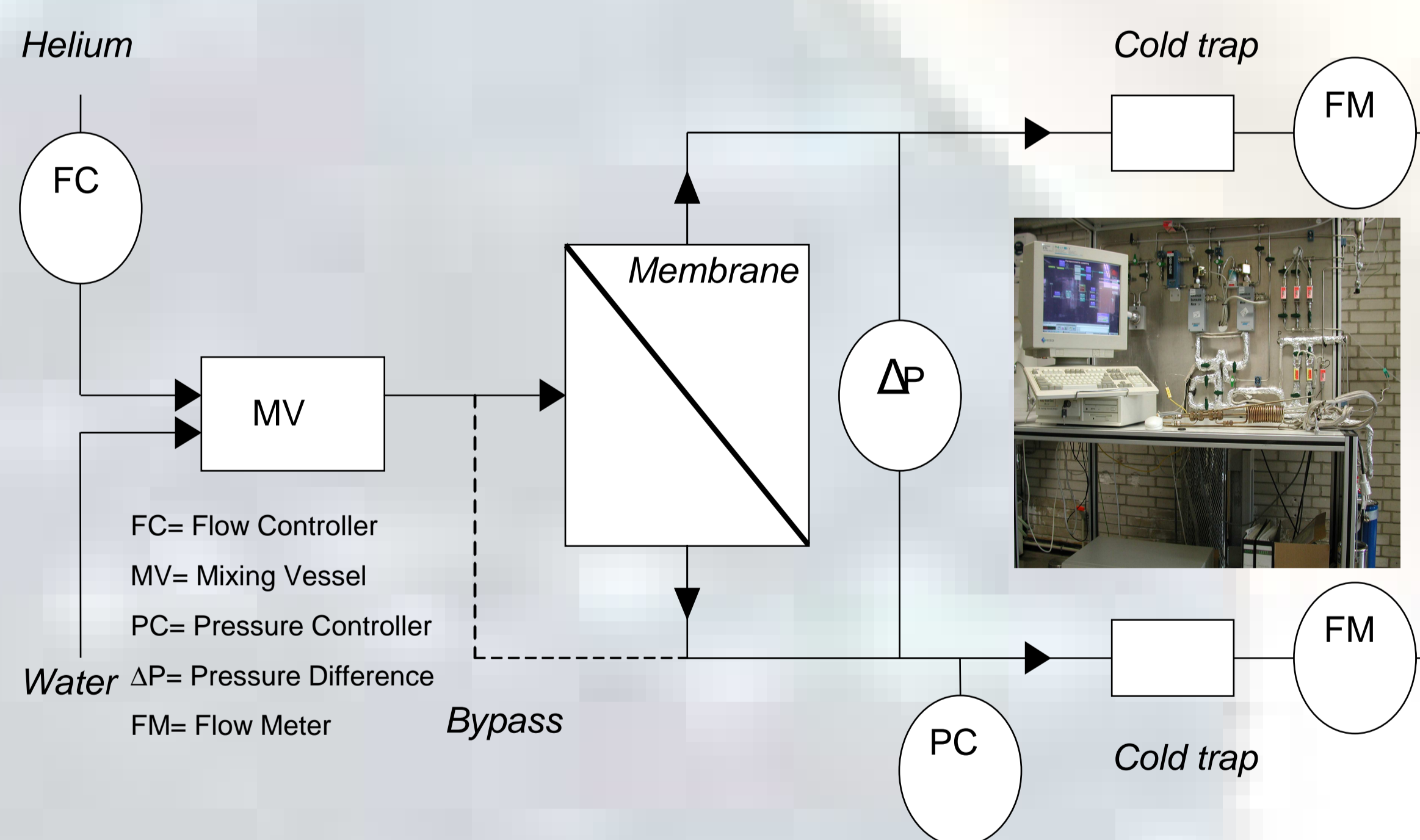


XRD patterns for γ -Al₂O₃, 25 mol% SiO₂ and pure SiO₂ calcined at 600°C for 3h in air

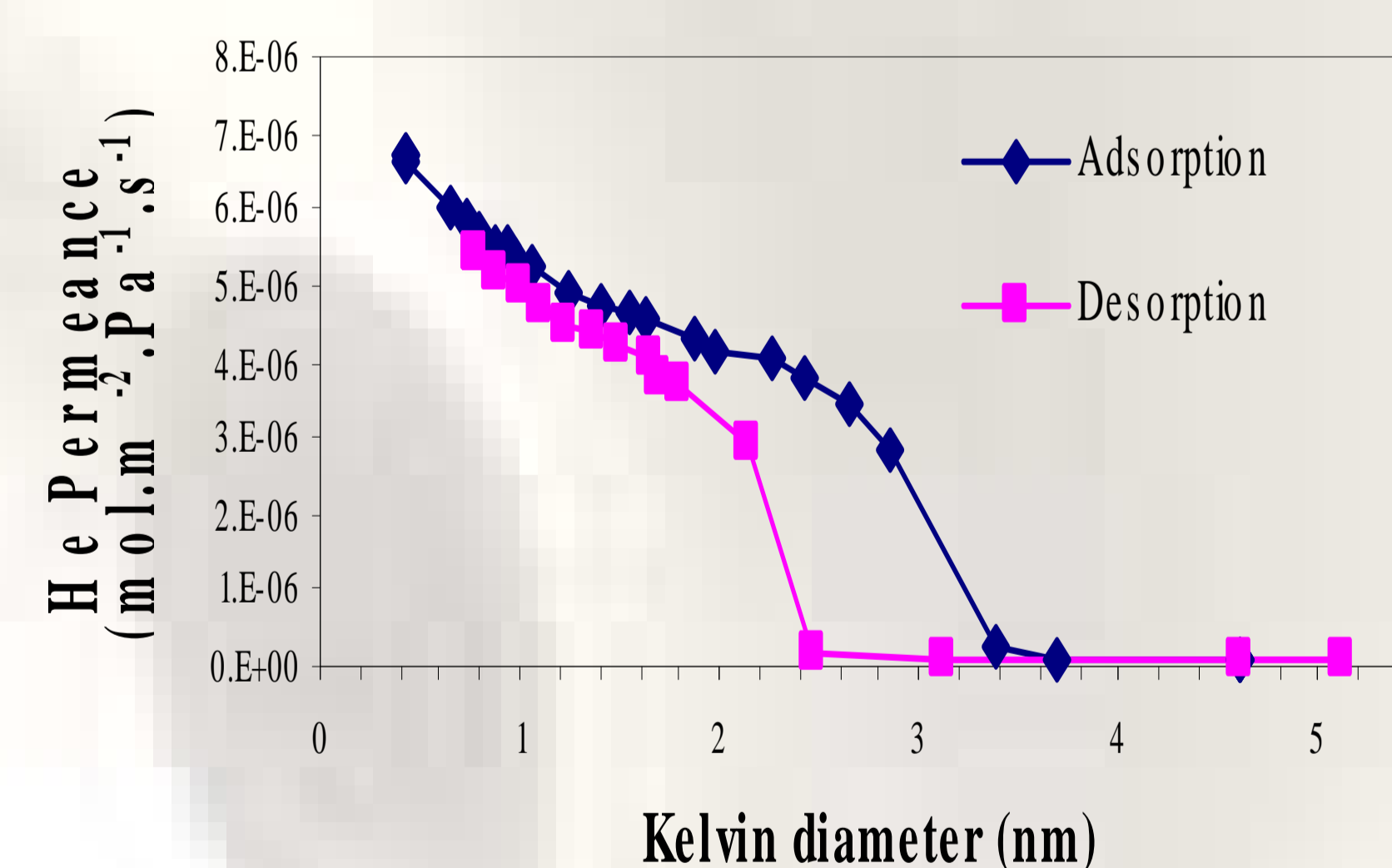


N₂ isotherms for γ -Al₂O₃, SiO₂ and composite thin films calcined at 600°C in air

Scheme of experimental permeometry set-up

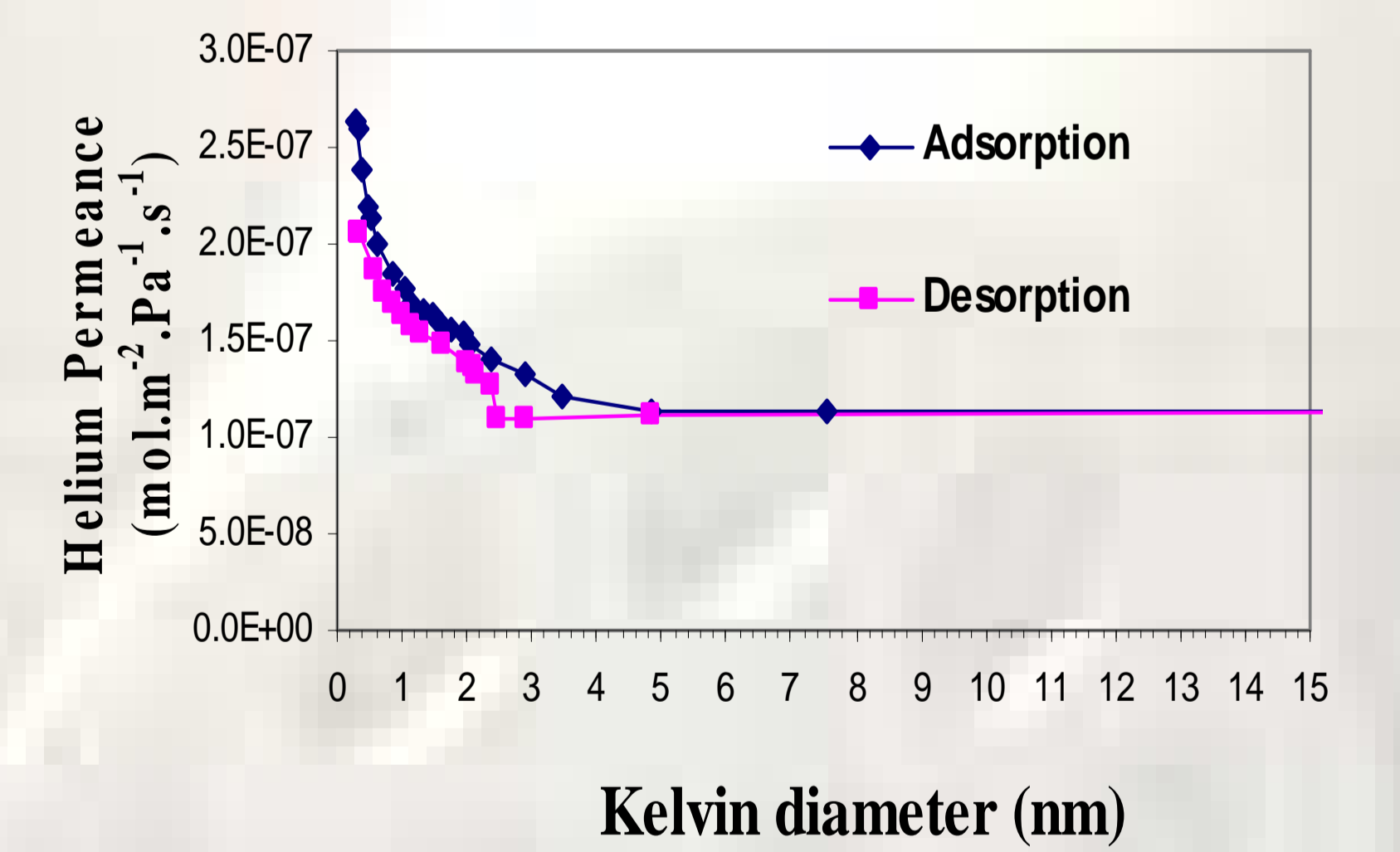


Pore size distribution for γ -alumina and composite thin films using the DFT model



Permporometry adsorption and desorption curves for supported γ -alumina membrane

Micro porous size distribution for composite thin films using the HK model



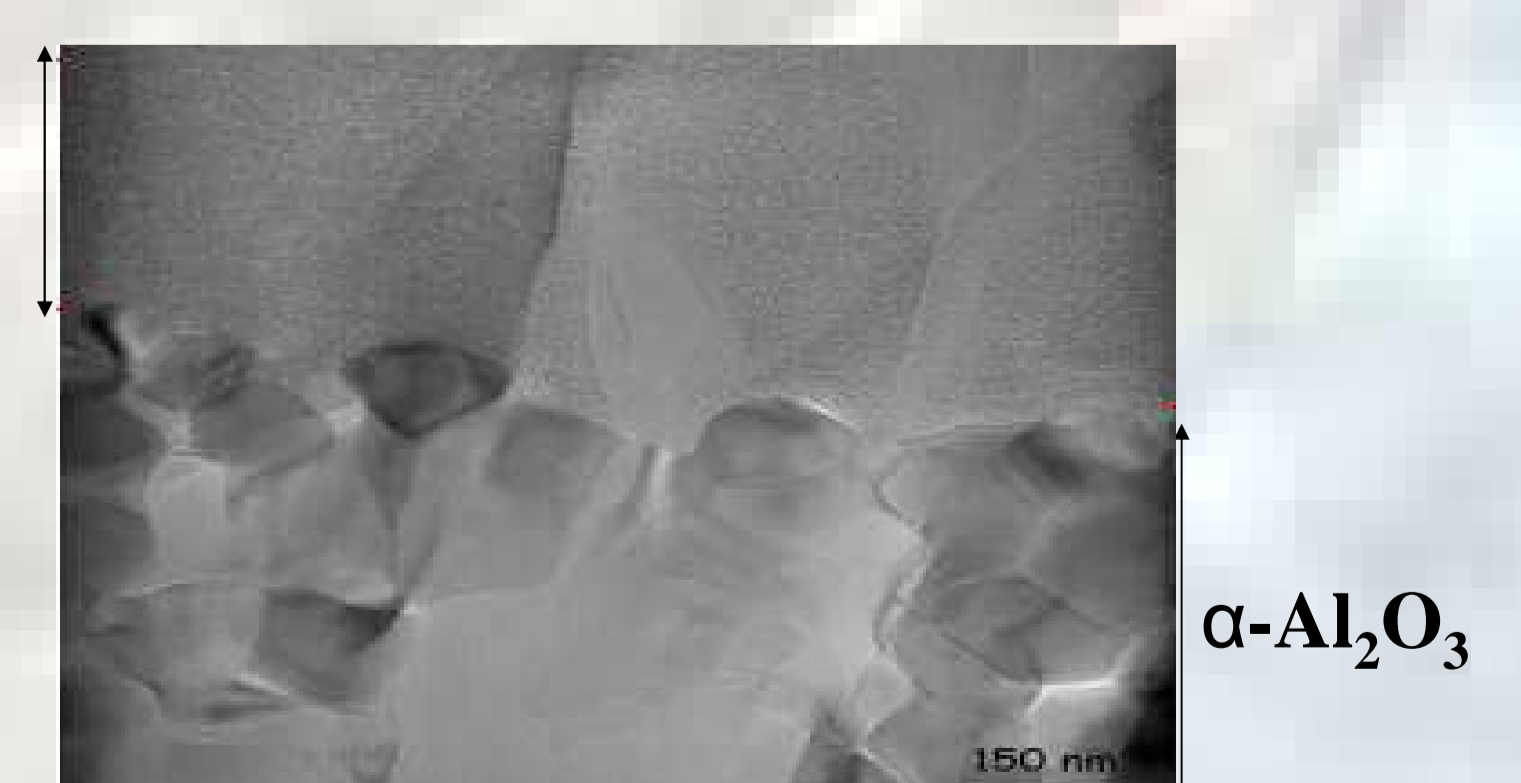
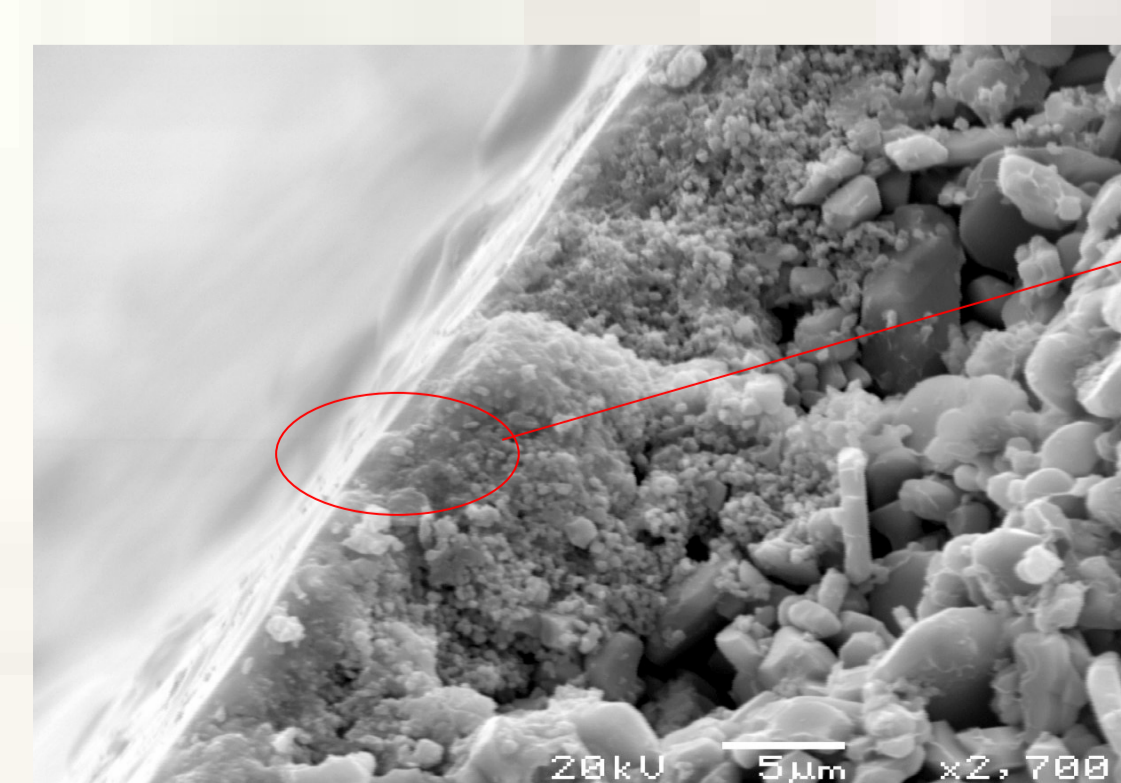
Permporometry adsorption and desorption curves of an alumina silica composite membrane with 55% mole SiO₂

Conclusions

- ✓ Supported γ -alumina membranes have a pore size of 2-3 nm without cracks or defects
- ✓ Composite Al₂O₃-SiO₂ thin films are mesoporous and microporous when silica mol% <36% and totally microporous when silica mol% >36% of the composition
- ✓ Microporous samples have a pore size of 0.5 nm

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SEM micrograph (left) and TEM micrograph (right) of the interface between the membrane and the macroporous cross section of a supported γ -Al₂O₃ membrane