Summary

This Ph.D. dissertation elaborates on the use of porous materials in fluid separation technology. Two different types of porous materials, namely surfactant templated silica and alumina have been used to prepare flat plate geometry porous membranes.

Two different synthesis routes of surfactant templated silica materials have been presented. Silica sol formulations have been taken from literature. Powders derived from sol 1 did not show any sign of an ordered mesoporous structure, however films derived from sol 1 on different supports did contain ordered mesoporous domains. It has also been shown that the ordered silica layer does not grow directly on the mesoporous substrate. Instead it grows on a structurally disordered silica-rich interface of thickness ~20 nm. Defect free layers could be formed from sol 1 both on macroporous and mesoporous supports. Powders and thin films grown on dense support, derived from sol 2, showed indications of an ordered mesoporous structure, but there was no sign of an ordered porous structure on porous substrates. Defect-free silica layers could be formed on mesoporous supports but not on macroporous supports. Water transport experiments showed that a silica layer deposited on an a-alumina support has the highest water permeability among all the silica layers, but the intrinsic permeability of the silica layer deposited on a-alumina support showed the lowest value. This is probably due to the microporous nature of the silica layer. Non-aqueous solvent permeation through the ?-alumina supported silica layer from sol 2 showed that hydrophilic liquids have a higher permeability coefficient than hydrophobic solvents.

Fluid flow through porous media receives considerable attention in this thesis. Three different pore sized ?-alumina membranes were prepared by calcining the membranes at three different temperatures namely 450?C, 600?C and 800?C. Fluid flow through porous media could be described in a very simple manner with Darcy’s law. It was found that the behaviour of all liquids (hydrophilic or hydrophobic) except water deviates from Darcy’s law (e.g. pressure vs. flux plot gives a positive x-axis intercept) below some critical average diameter of the porous medium. The critical pore diameter is not the same for each and every liquid. To understand this behaviour we took into account two structural factors, namely porosity and tortuosity of the porous medium, which can affect the permeability of the liquids, and a physical parameter, namely the viscosity of the liquids. It has been found that the presence of water as a secondary minority species in the solvents plays an important role in the mechanism of solvent transport through porous media. Based on the observed phenomena and avail-

able literature a hypothesis of liquid transport through porous media has been proposed.

The recovery of an industrially important poly-oxo-metalate (POM) catalyst using two types of ?-alumina membranes is also discussed. Nanofiltration experiments were carried out on aqueous solutions of $Na_{12}POM$ and an organic liquid mixture containing $Q_{12}POM$ (Q=quarternary ammonium ion). The ?-alumina membrane calcined at 600°C showed > 96% retention of POM ions, regardless of the nature of the counter ions and the nature of the solvent. ?-Alumina membranes calcined at 900°C showed a lower level of retention for toluene soluble $Q_{12}POM$. The analysis showed that the retention of the catalyst is due to both double layer overlap and size exclusion for both water-soluble $Na_{12}POM$ and toluene soluble $Q_{12}POM$. Liquid permeability studies showed that aqueous POM solutions obey Darcy flow behaviour, while a non-zero threshold pressure needs to be exceeded before (non-aqueous) hydrophobic liquid transport takes place. This threshold pressure was absent for ?-alumina membranes calcined at 900°C, and this is most likely due to the larger pore size in the latter system. NMR and IR investigations on the final reaction mixture showed that the POM catalyst is stable under the used reaction and permeation conditions. XPS measurements together with experimental data showed that the POM did not enter the pores of the membrane, and thus it could be recovered easily. It has also been shown that the catalytic activity of the POM moiety increased with the number of membrane recycles.