Functionalization of ceramic membranes by polymer grafting for solvent nanofiltration.

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Presently, large interest in Nano Filtration (NF) is being devoted to filtration of organic solutions. However, instead of using the established NF membranes for aqueous applications, solvent resistant nanofilration (SRNF) membranes are required. The main challenge for further expansion of SRNF membranes is the development of membranes that are stable in a wide range of organic solvents, as well as showing reproducible and long term high performance (i.e. high fluxes and enhanced selectivity).

Since it is generally accepted that inorganic materials, as alumina, titania and zirconia are inherently more stable than polymers (i.e. excellent structural integrity, mechanical, thermal and chemical stability in all types of polar and non-polar organic solvents without problems of swelling and compaction), they are a promising alternative for the current polymeric membranes. However, due to the presence of surface hydroxyl groups these inorganic materials show hydrophilic behavior limiting, their application for non-aqueous solutions. In order to overcome this limitation, polymer chains are terminally and covalently bonded onto the surface and inside the pores of a porous ceramic support. The grafted polymer imparts the desired membrane selectivity while the ceramic support provides the mechanical, thermal and chemical stability.

Polysiloxanes, like PDMS, present a unique combination of characteristics (e.g. thermal and mechanical stability, hydrophobicity and solubility in non-polar solvents). However, their swelling behavior hampers its application. The introduction of a covalent bond between a ceramic support and polymer, as well as the confinement of the polymer in the pores, is expected to decrease swelling and to increase membrane affinity.

In this work, the development of a PDMS grafted γ -alumina membrane (5 nm) is described. Surface modification is based on the formation of an aminosilane layer (APTES) on a hydroxyl-terminated substrate and subsequent reaction of the amine group with an epoxyterminated PDMS. This grafting reaction is schematically given in the figure below.



Silane-grafted alumina + epoxy-terminated PDMS \rightarrow PDMS-grafted alumina

The influence of several reaction parameters on the pore size/pore structure and membrane performance is evaluated. FTIR and TGA results confirmed that both linker and polymer were successfully grafted onto alumina flakes (5nm). Gas adsorption measurements showed that both surface and pore walls were successfully modified. Contact angle (θ) measurements demonstrated that hydrophobic grafted membranes were fabricated.



 $\theta = 94 \pm 1^{\circ}$

Permporometry confirmed that the pores walls were successfully grafted. Solvent permeation showed that reproducible, stable, high solvent flux nanofiltration membranes were fabricated.

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