Ceramic NF membranes with a cut-off of 450 Da have been already developed 10 years ago [1]. These membranes were intensively tested for the cleaning of hot waste water of textile finishing close to the washing machine [2]. A first plant containing 25 m$^2$ was installed in 2002 which is still in operation without membrane replacement demonstrating the outstanding stability of the membrane. A few years later the fabrication of this membrane was fully commercialized by the Inopor GmbH. A lot of different applications have been realized in waste water treatment, product cleaning, biomass treatment as well as catalyst recovery [3]. But in some tested applications the cut-off of the membrane was not sufficient or the flux of the membrane dropped down quickly because of pore blocking.

New developments were focused on a further reduction of the cut-off below 450 Da. Membranes were prepared by polymer sol-gel technique starting from a mixture of titanium-isopropoxide and zirconium-n-propoxide. The hydrolysis of the alkoxides was controlled either by the amount of water and/or the addition of protective groups. The coating of tubes was performed in a cleanroom (class 100) under controlled humidity. Final thermal treatment was performed at a temperature between 300°C and 500°C in air as well as under inert atmosphere. Final membranes were characterized by permporometry and filtration tests using polyethylene glycols (PEG) as molecular standards.

High quality membranes were obtained on high quality substrates with a final intermediate layer of zirconia with a pore size of only 3 nm. On monochannel tubes with an O.D./I.D.=10 mm/ 7 mm a molecular cut-off (MWCO) in aqueous solution of polyethylene glycols of 210 Da was achieved with a specific flux of 9 l/(m$^2$h/bar) at a transmembrane pressure of 30 bar. The pH-stability was tested by filtration of 1M, 2M, 4M HCl as well as NaOH solution for 2 hours. The MWCO measured before and after this exposure remained constant. Upscaling of membrane preparation to long multichannel tubes is in progress. The development is part of a big national project. The funding of the project by the Federal Ministry of Education and research (FKZ: 03X0080L) is grateful acknowledged.

References:


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