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# Preparation and characterization of novel titania-modified ceramic membranes

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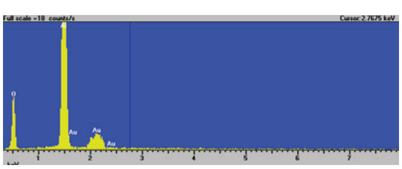
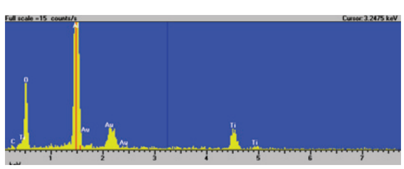
Membrane processes have gained a great deal of attention during the last years, in order to replace the conventional energy intensive methods (e.g. cryogenic distillation). Extensive R&D has been conducted both in gas and liquid separation processes. One of the limitations for a massive industrial penetration of membranes is the lack of suitable membrane materials capable to withstand the harsh industrial conditions for a prolonged period of time [1]. This is because the majority of membrane materials that have been commercialized up to now are polymeric. On the other hand, microporous ceramic membranes have great potential for opening up new emerging application fields in processes where polymeric membranes can not be applied. Ceramic membranes have the advantages of unique chemical and thermal stability, high mechanical strength and long lifetime compared to polymeric membranes. Of course these advantages are dependent on the type of materials used for the preparation of ceramic membrane [2]. The most well-studied microporous ceramic membranes in the literature are the asymmetric alumina/silica membranes. Nevertheless, reproducibility and stability (at high temperatures, $H_2O$ vapours and at strong acidic conditions) problems of these membranes hinder their commercialization especially for gas separation applications. Recently, the scientific interest regarding ceramic membranes turned mainly to microporous titania membranes because they seem to be more stable than silica membranes, while their photocatalytic activity opens new fields of application including e.g. the photocatalytic wastewater treatment or even the photocatalytic water splitting for $H_2$ production [3, 4].

We have recently developed methods for preparation of high-quality microporous silica membranes by controlling their pore size with vapour phase deposition techniques [5, 6, 7]. In this study we attempt to apply these methods for the preparation of novel titania-modified microporous ceramic membranes, which seem to be much more attractive for various applications. Titanium-isopropoxide was used as titania precursor and $O_2$ or $O_3$ as oxidizing agent in the modification experiments. Two different types of membranes were used as starting materials in the modification experiments: a) hollow fiber silica membranes, b) disc-shaped γ-alumina membranes. The membranes were characterized with various methods before and after modification. Their gas separation performance was evaluated with single gas permeation experiments, their porous structure was evaluated with the permporometry method and finally morphological and structural observations were done with SEM/EDX.

All the methods that tested so far proved effective for the deposition of titania on the membrane surface. Figures 1 and 2 show the EDX spectrum images of a γ-alumina membrane before and after 3 C-ALD cycles (alternating exposure to titanium-isopropoxide and $O_3$) and a silica membrane before and after 4 hours of CVI modification (simultaneous feed of titanium-isopropoxide and $O_2$) respectively. The emergence of titanium peaks, between 4.5 and 5 keV in the spectrum, after the modification (Figures 1 and 2 (b)) confirms the titania deposition.



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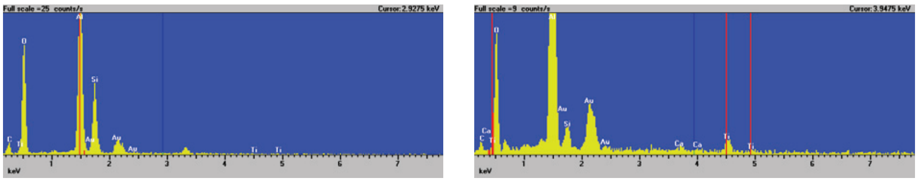
(a) (b)

Figure 1: EDX images of a γ-alumina membrane before (a) and after (b) C-ALD modification.



(a) (b)

Figure 2: EDX images of a silica membrane before (a) and after (b) CVI modification.

The respective SEM images did not show any noticeable change on the membrane surface, indicating that the created deposits were in the nano-scale. The titania deposition was also confirmed by the gas permeation and permporometry experiments, which showed decreased permeance and pore size respectively, after the modification processes. Further efforts will be focused in optimizing the membrane performance (depending on the specific application that the membranes are indented to be used) by controlling the deposition variables of titania.

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Keywords: titania membranes, microporous ceramic membranes, ceramic membrane modification