Preparation of palladium-coated α-alumina tubular membranes by sol-gel and electroless plating

T. Bisoto[a], W. P. Silvestre[a], V. Santos[b], M. Zeni[C] and C. Baldasso[a,*]
[a] Postgraduate Program in Process Engineering and Technologies (PGEPROTEC), University of Caxias do Sul, Caxias do Sul, Brazil;
[b] Postgraduate Program in Mining, Metallurgical and Materials Engineering (PPG3M), Federal University of Rio Grande do Sul, Porto Alegre, Brazil.
[C] Federal Institute of Education, Science and Technology of Rio Grande do Sul, IFRS
[*] cbaldasso@ucs.br

Abstract

Using composite inorganic membranes in gas separation has become a promising alternative, with low cost and smaller energy consumption. This work aimed to assess the feasibility of producing Pd-coated α-alumina tubular ceramic membranes for gas separation. The metallic deposition was performed using the sol-gel and electroless plating methods, promoting the reduction of the ions Pd^{2+} to metallic Pd to form the selective layer. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) characterized the ceramic supports with and without the Pd layers. The SEM and EDS analysis confirmed the palladium deposition on the membranes, indicating that the sol-gel and electroless plating are suitable methods to produce Pd-coated membranes based on α-alumina supports.

Introduction

Membrane-based technologies are currently an important research topic in separating gas mixtures [1]. Such processes are important to reduce greenhouse gas emissions to the atmosphere. Global warming is associated with fossil fuels, which deplete fossil energy reserves, a problem that has encouraged using renewable energy sources, such as hydrogen [2]. Within this context, membrane separation processes became an attractive alternative with a growing body of research. Inorganic membranes have been widely used in gas permeation and separation in chemical and biotechnology industries [1].

When coated with palladium, ceramic supports combine the metallic layer's high selectivity with the porous support's high permeability [3]. The main applications of these types of membranes are the catalytic recovery of hydrogen and the separation of carbon dioxide and methane separation [1]. Since the gas permeation is inversely proportional to the thickness of the palladium layer, extremely thin palladium layers are necessary to achieve higher permeate fluxes [4].

The sol-gel and the electroless plating methods can carry out the deposition of the palladium layer on tubular ceramic supports. The sol-gel technique promotes uniformity and purity of the support particles, avoids possible cracks on the metallic layer, and prepares the support to adhere to the metal to be deposited. The metallic electroless deposition consists of a chemical reduction or autocatalytic reaction of the metallic ions that will be deposited on the substrate that just went through the sensitization and activation steps. The coating forms without electrodes or electrical currents [3,5].

Within this context, this work aimed to promote the deposition of a palladium layer on ceramic membranes using the sol-gel technique associated with the electroless plating and characterize the efficiency of the deposition with the morphological characterization by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) of the developed membranes.

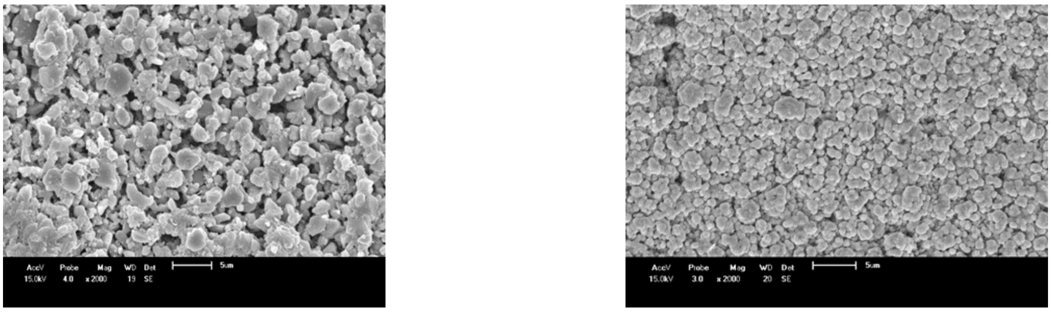
Materials and methods

All membranes in this work were prepared via the sol-gel and electroless plating techniques according to the methods described by David and Kopac [3] and Sari et al. [5-6].

The morphology of the α-alumina support and the coated membranes were studied by SEM and EDS using a Jeol (JSM - 5800) microscope. The samples were sputtered with gold before analysis.

Results and discussion

The SEM micrographs of the α-alumina support layer and the membranes produced by Pd coating are shown in Fig. 1.

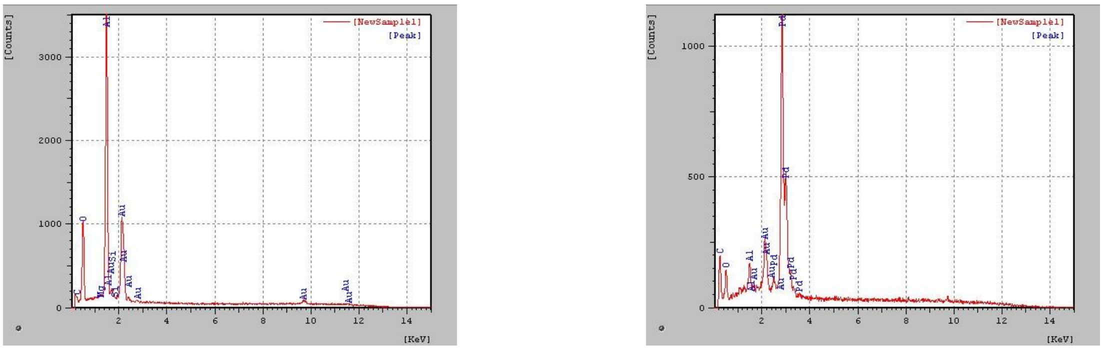


(A) (B)

Fig. 1 – SEM micrographs of the α-alumina support (A) and the membrane produced with one layer of Pd coating produced by the sol-gel and electroless plating methods.

Fig. 2(a) presents the surface of ceramic support without Pd coating and shows a porous structure with imperfect grains. This same morphology is reported in the literature and is characteristic of α-alumina supports. Fig. 2(b) shows the membrane surface after Pd deposition. It is possible to observe that the surface was covered with Pd, with a visible decrease in the size and number of pores.

The EDS graphs of the α-alumina support layer and the membranes produced by Pd coating are shown in Fig. 2.



(A) (B)

Fig. 2 – Results of EDS analysis for the α-alumina support (A) and the membrane produced with one layer of Pd coating produced by the sol-gel and electroless plating methods.

The ceramic support (Fig. 3(A)) showed strong signals of α-alumina and Au, evidenced by the higher peaks of Al (representing alumina), followed by Au (gold), which was present due to sample sputtering. In Fig. 3(B), the coated membrane had strong Pd signals, with alumina and Pd peaks, confirming the palladium deposition on the membrane surface. The much smaller signals of Al indicate that Pd coated most (if not all) of the support surface.

Conclusion

The morphology of the membranes showed that all had a uniform distribution of the Pd coating throughout the external surface, and the coating thickness and microstructure images showed that this technique was suitable for producing palladium-coated α-alumina tubular membranes.

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