DEVELOPMENT OF NUCLEAR TRACK MEMBRANES IN THE ULTRAFILTRATION RANGE USING THE IEA-R1 NUCLEAR REACTOR

I.M. Yamazaki, L.P. Geraldo

Instituto de Pesquisas Energeticas e Nucleares - IPEN/CNEN-SP
C.P. 11049 Pinheiros, 05422-970, Sao Paulo (SP), Brazil

ABSTRACT

In this paper, a new experimental apparatus for porous membrane production, in the ultrafiltration region and using the fission track registration technique in Makrofol KG (10 μm thickness) is described. The irradiation device has been installed near the IEA-R1 (2 MW) nuclear reactor core, at a position where the thermal neutron flux is around $5x10^{10}$ $n/cm^2.s$. Filtering membranes with pore diameters in the range from 0.1 to 0.03 um and density around $8x10^8$ pores/cm^2 to $1.5x\ 10^9$ pores/cm^2 were produced employing irradiation times of 3 to 6 minutes. A preliminary calibration curve pore diameter versus etching time is presented.

INTRODUCTION

Microfiltration and Ultrafiltration membranes are routinely used in several applications in the Industry and Biotechnology such as: in MF: clarification of fruit juice, beer and wine; waste water treatment; air filtration; water sterilization (viruses, bacteria), etc. and in UF: potabilization of water; treatment of effluents in the textile industry; recovery in electro-dip-coat painting; dairy industry (concentration of milk and cheese whey), etc.These membranes are used to filter particle sizes from 10^3 Å (0.1 μm) to 10^5 Å (10 μm) and 20 Å (0.002 μm) to 10^3 Å (0.1 μm), respectively and there are many companies competing in this market nowadays.

Our Laboratory has concluded recently the development of microfiltration track etch membranes [1,2], with porosity between 10^5 to 10^8 pores/cm^2, using the Solid State Nuclear Track Detection (SSNTD) technique and in this work, a new irradiation apparatus constructed near the core of the IEA-R1 swimming pool type nuclear research reactor, in order to develop UF track etch membranes is presented. By means of this new experimental device, we obtained filters with pore sizes from 300 Å (0.03 μm) to 1000 Å (0.1 μm) with similar characteristic to commercially known membranes [3].

EXPERIMENTAL METHODS AND RESULTS

The experimental method consists of exposing thin films to a collimated beam of fragments produced by the U-235 fission with thermal neutrons coming from the core of the IEA-R1 reactor. These fission fragments produce tracks across the entire thickness of the plastic foil which may be selectively attacked (etched) by an appropriate chemical solution, producing a fine channel or hole in the bombarded material. The hole diameter is controlled by the etching time and the density of hole is determined by the particle dose or irradiation time [4].

The irradiation device for porous membrane production is presented in Fig. 1. It consists of a evacuated aluminium chamber which in its central position is fixed an aluminium rod (4 mm diameter) where a fissile material, U_3O_8 93.15% enrichment, is deposited. The Makrofol KG foil is then positioned around the fissile material, in the inner wall of the irradiation chamber. After that, the chamber is placed inside an irradiation guide tube (Fig. 2) where the thermal neutron flux is around $5x10^{10}$ n/cm^2.s. By means of a long aluminium tube (5/8" diameter, 10 m length) (Fig. 1 and 3), evacuation in the whole apparatus is made until 10^{-3} torr pressure is reached. The radioactive gases released during the fission reaction are captured in a glass trap which is kept in nitrogen liquid temperature during all irradiation period.

Irradiation time from 3 min. to 6 min. was adequate for obtaining membranes of $8x10^8$ pores/cm^2 to $1.5x10^9$ pores/cm^2. After irradiations, Makrofol KG films were developed (etched) in 5 N NaOH solution, 35 °C temperature, in etching time from 5 to 30 min. and the pore diameters and porosities were analysed by Scanning Electron Microscope (SEM).

In Fig. 4 is showed a preliminar calibration curve, pore diameter versus etching time, for track diameter from 0.03 μm to 0.1 μm and pore density of $8.3x10^8$ pores/cm^2. This curve, in the ultrafiltration range, did not present a linear behavior as presented in the microfiltration range and this result is in good agreement with other authors [5,6]. In addition, some SEM microphotographies are also showed in Fig. 5 and 6, with average pore size of 0.065 μm and 0.045 μm, respectively, with a magnification of 40,000 and, in conclusion, additional studies are being carried out in order to obtain track etched membranes with pore sizes less than 0.03 μm.

ACKNOWLEDGEMENTS

The authors thank Mr. Celso V. de Moraes, from MMQ-IPEN/SP, by preparation of samples for SEM analysis and to Mr. Roberto J. Rodrigues, from LSI-EPUSP, by his assistance during the utilization of SEM.

REFERENCES:

1. Yamazaki, I.M. and Geraldo, L.P. Proc. Workshop CEE-Brazil on Membrane Separation Processes, Angra dos Reis, RJ, may 1992, p. 125.

2. Yamazaki, I.M. and Outi, E.Y. Paper to be published in Int.J.Radiat. Appl.Instrum. Part D. Nucl. Tracks Radiat.Meas., 1994.

3. Nuclepore Corporation catalogue. Products for laboratory process filtration. Pleasanton, California, 1988.

4. Fleischer, R.L.; Price, P.B.; Walker, R.M. Method of forming fine holes of near atomic dimensions. Rev.Sci.Instrum. 34(5):510-12, 1963.

5. Trautmann C.; Spohr, R.; Vetter,J. Controlled Production of Submicron Pores. Darmstadt, Gesellschaft fuer Schwerionenforschung, m.b.H., 1988, GSI 88-1, p. 246.

6. Heyna, U., Diplomarbeit, 1985. APUD Trautmann C.; Spohr, R.; Vetter, J. Controlled Production of Submicron Pores. Darmstadt, GSI 88-1, p. 246.