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Processing and Characterization of Microfiltration Supports Prepared from Alumina Powders

Gamze Gül Avcı,[a] Zülal Mısırlı[a] & Volkan Günay[a,b]

[a]TÜBİTAK-MRC, Materials Department, PO Box 21, Gebze, Turkey
[b]Istanbul Technical University, Department of Metallurgical Engineering, Maslak-İstanbul, Turkey

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Abstract: Alcoa A-16 SG commercial alumina powder is widely used for the preparation of porous supports for alumina membrane layers. In this work, a comparison is made concerning the use of different alumina powders. For this purpose two types of powders with different physical and chemical characteristics to Alcoa powder were chosen. The powders of interest were Seydişehir alumina, which is a local alumina produced for metallurgical purposes by the Bayer process, and the granulated form of this alumina obtained by spray-drying. Due to the coarser microstructure of the substrates prepared from spray-dried powders, the pore size was larger and the amount of the flux was higher than the other two substrates which were prepared from the processed Seydişehir and Alcoa powders in identical conditions.

1 INTRODUCTION

Most ceramic membranes have a composite structure, consisting of two or more layers. One of the layers is the support which usually gives the required strength to the membrane and also should have certain separation ability with appropriate pore size and pore-size distribution. The porosity in each membrane layer consists of a well-controlled size range to provide the desired separation selectivity. Although ceramic membranes can be used in fine filtration (2–10 μm), most of them are used in microfiltration (0·02–2 μm) and ultrafiltration (0·001–0·02 μm) applications. The top layers of the membrane are usually prepared by coating the substrate using the sol–gel technique.[1] Alcoa A-16 SG commercial alumina powder is widely used for the preparation of the porous supports. In this work, Alcoa A-16 SG and Seydişehir alumina, which is the electrofiltration residue of a local alumina, produced for metallurgical purposes by the Bayer process, were used. Seydişehir alumina was used in two forms; (1) it was spray dried after calcination at 1100°C for 1 h and (2) its chemical properties were improved by burning off the carbon impurities at 600°C, then washing in warm water to reduce the Na content, followed by calcining at 1200°C for 1·5 h and grinding.[2,3] The properties of the powders used in the present work are given in Table 1.

2 EXPERIMENTAL

The substrates were prepared using spray-dried Seydişehir powder (SDSP),[3] processed Seydişehir powder (PSP) and commercial Alcoa A-16 SG powder (AP), by uniaxial dry-pressing in disc shapes with a diameter of 40 mm and a thickness of 2·5 mm. Pressing pressure was around 100 MPa. The disc-shaped samples were sintered at 1200 and 1400°C with a heating rate of 1°C/min. The sintered samples were characterized by measuring density, porosity and pore size by using an Hg-porosimeter (Micromeritics Autopore 11 9200). Sintered (or pre-sintered) substrates were then coated using 1 M Boehmite sol which was prepared from Al-tri-sec butoxide[4,5] and the coated membrane configuration was heat-treated

Table 1. Properties of the alumina powders

Powder	Density (g/cm^3)	Particle size (μm)	Surface area (m^2/g)	Na content (%)
Original Seydişehir	3·49	3·6	41·7	0·8
SDSP	3·63	8·4	6·2	0·8
PSP	3·94	5·6	5·0	0·2
Alcoa A-16	3·95	0·38	9·0	0·06

Table 2. Properties of substrates sintered at 1200°C

Substrate	Relative density (%)	Porosity (%)	Aver. pore diam. (μm)
SDSP	49·8	50·4	0·2185
PSP	51·1	48·9	0·1518
AP	67·7	32·3	0·0986

at 600°C for 3 h. The water flux was determined in a home-made pressure vessel using ultrapure water. The micromechanism and the microstructure of the substrates and the membrane layers were examined by using scanning electron microscope facilities (Jeol JXA 840 A).

3 RESULTS AND DISCUSSIONS

The average pore diameters of the substrates are given in Tables 2 and 3. The substrates prepared from SDSP have larger pores than from PSP which is the result of increased particle size of the powder due to the granulation during spray-drying. Sintering at 1400°C decreases the porosity of both substrates while increasing the pore size even more.

Water flux measurements were done on SDSP, PSP and AP substrates sintered at 1200 and 1400°C with membrane top layers calcined at 600°C for 3 h, up to a pressure of 10 bar. Flux measurements (Fig. 1) show that the water flux

Table 3. Properties of substrates sintered at 1400°C

Substrate	Relative density (%)	Porosity (%)	Aver. pore diam. (μm)
SDSP	54·3	45·7	0·2499
PSP	59·6	40·4	0·1609
AP	80·5	19·5	0·1052

increases with increase of the pore size of the substrate for a membrane top layer having the same pore size due to standard calcination temperature. Usually pressures up to 5 bar are sufficient in microfiltration. No cracks or bursting of the AP and PSP substrates were detected during the flux measurements with pressures applied up to 10 bar, but owing to the high porosity of the SDSP substrates, the strength is low and after about 7.5 bar, cracks occurred. Sintering the substrates at 1400°C decreases the porosity to a level sufficient to withstand pressures up to 10 bar without any negative effect on the amount of the flux. It is clearly seen in Figs 2 (a) and (b), that the membrane top layers on SDSP and PSP substrates are homogeneous.

With the SEM visual examination, the fracture surfaces of these samples have been identified and described in terms of microstructural parameters such as pore and grain distributions and the effect of the substrate grain size. There is a good bonding between the substrates and the top layers as shown in Figs 3 (a)–(d). This is also true for the SDSP substrate which has higher porosity and larger grain size than the SPS and AP substrates. It can be seen in Fig. 3 that the membrane top layer thickness is dependent on the porosity of the substrates. The higher the porosity in the substrates, the thicker the membrane top layer. The microstructures show that the SDSP substrate has larger particles than the PSP substrate and shows coarser microstructure.

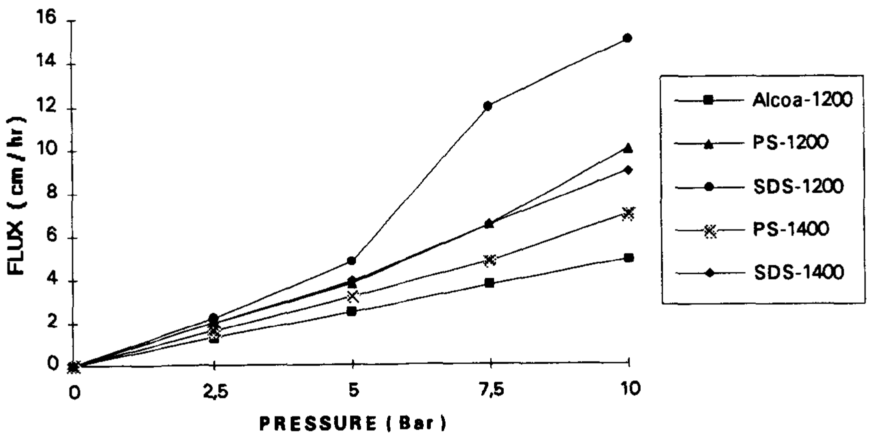

Fig. 1. Water flux of the systems sintered at 1200 and 1400°C.

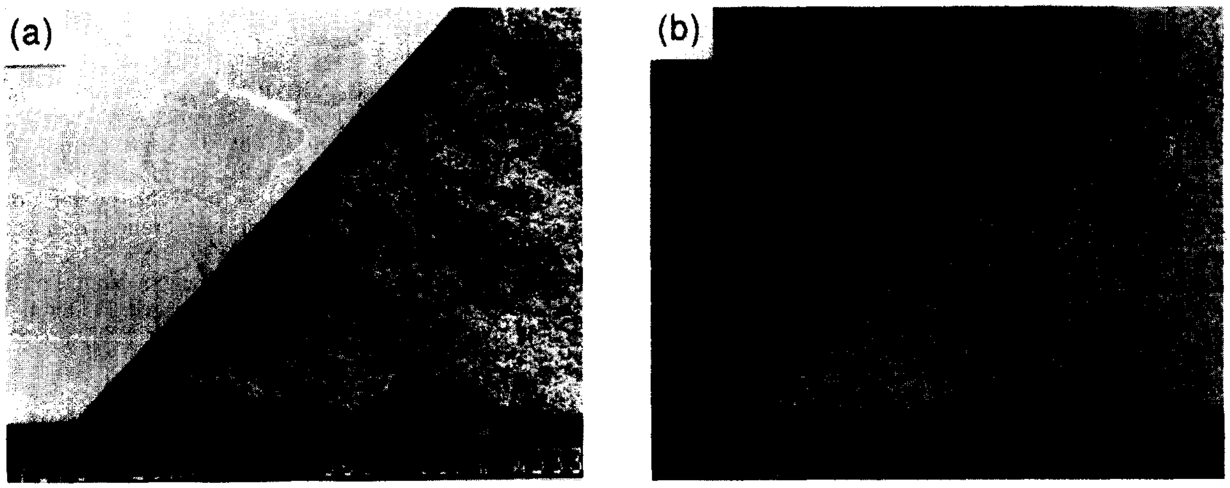

Fig. 2. Membrane top layers on (a) SDSP, (b) PSP substrates.

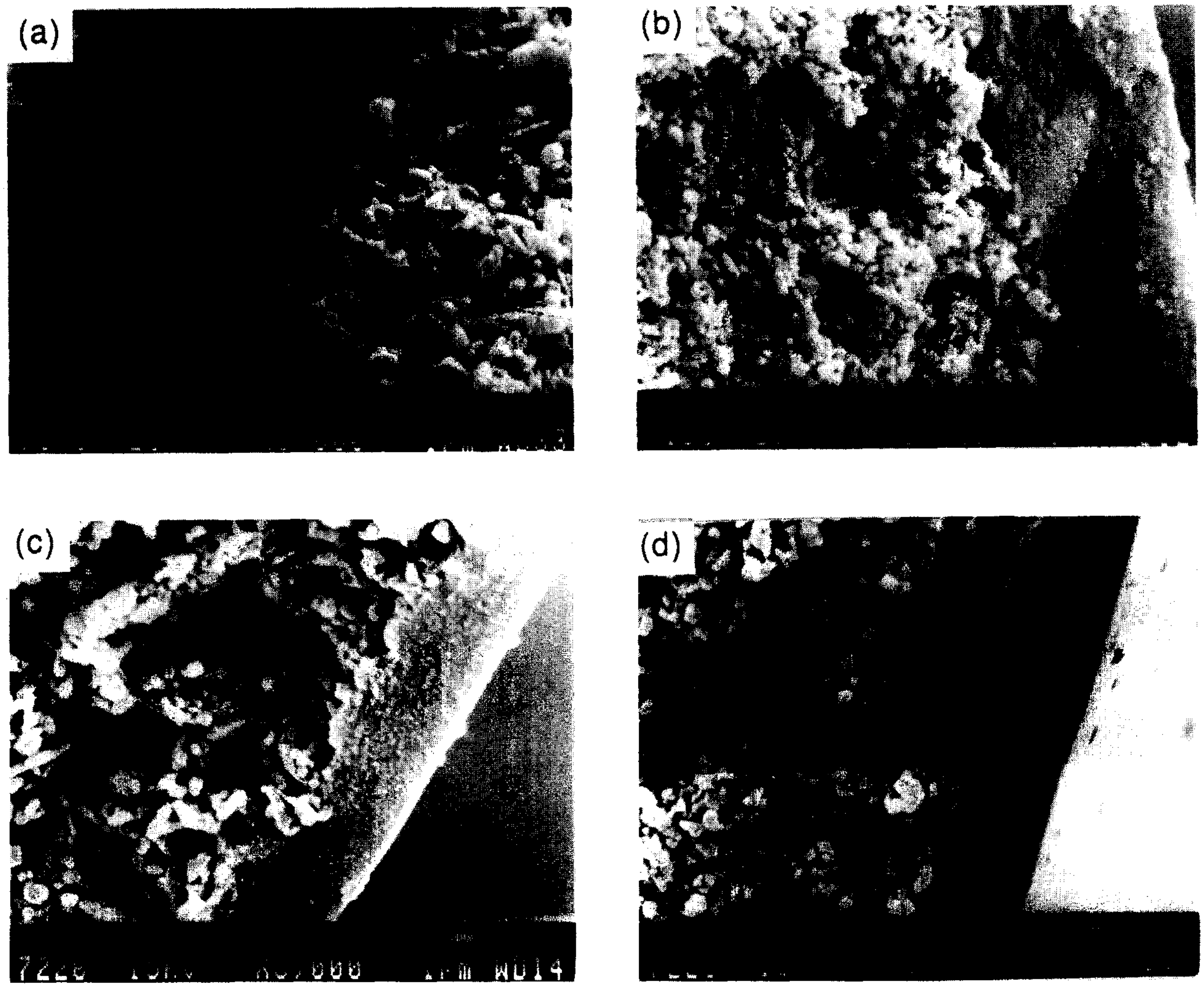

Fig. 3. SEM micrographs of (a) SDSP-1200°C, (b) PSP-1200°C, (c) SDSP-1400°C, (d) PSP-1400°C.

4 CONCLUSIONS

Porous supports for ceramic membranes can be prepared from the electrofilter residue of metallurgical grade alumina (after some processing steps) by dry-pressing and sintering at 1200 and 1400°C.

The supports obtained can be used in microfiltration processes but have to be evaluated for high pressures.

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