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Permeation and Separation Characteristics of Supported Alumina and Titania Membranes

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ABSTRACT

Preparation, characterization, water permeability, and filtration characteristics of alumina and titania membranes on alumina and on clay supports are described. Commercial α-alumina and Gangetic silt have been used for the preparation of support disks. The effects of temperature and time of sintering on porosity and pore size distribution of the supports have been studied. A higher firing temperature reduces the porosity but increases the average pore diameter. The sol–gel technique has been used to prepare supported membranes of alumina and titania. Stability of the sols prepared was noted, and some of the sols were found to remain stable for about a year. Coating and firing cycles had to be repeated in order to get good membranes. However, SEM pictures show that cracks developed in membranes if more than five coatings were applied. The membranes had good water permeabilities. The rejection performances of the membranes were studied using bovine serum albumin of 66,000 dalton molecular weight. The dependence of the solute rejection on the coating and firing cycles for different sol concentrations was measured.

INTRODUCTION

Industrial applications of inorganic membranes, particularly ceramic membranes, for separation activities like ultrafiltration, nanofiltration, wastewater treatment, and gas separation are now well-established. The advantages that this class of membranes offer over their polymeric counterparts are also well-documented (1). Most of the ceramic membranes available in the market or developed in the laboratory are of the composite type with a permselective ul-

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trathin membrane layer coated on a macroporous substrate. The substrate or support is generally made of alumina or carbon, although other materials like clay have shown considerable potential.

In recent years there has been substantial progress in the preparation of membrane supports and supported membranes. The research thrust has been on the preparation of membranes having controlled pore sizes distributed over a narrow range. Special emphasis has been placed on attaining pore sizes of the order of nanometers or less for applications in the separation of gases or low-molecular-weight solutes from solutions. Peterson et al. (2) reported the preparation of alumina-supported titania membranes by the sol–gel technique starting with the precursor 2-methyl-2-butoxide, and could attain pores small enough to give a molecular weight cutoff as low as 200. The average pore diameter was about 10 nm. De Lange et al. (3) also used the sol–gel technique to prepare γ-alumina membranes on an α-alumina support by dip coating. They modified the membranes thus prepared by applying a further coat of silica using a silica sol. They also prepared supported membranes using binary sols of titania, zirconia, and alumina in combination with silica. The mesoporous membranes produced were tested for separation of a mixture of H_2 and CH_4. In a similar study, Ohyo et al. (4) prepared tubular supported membranes using a mixed sol of zirconia, silica, and yttria for use in H_2 gas separation. Vroon et al. (5) prepared defect-free zeolite membranes, about 5 nm thick, on porous-alumina supports by in-situ crystallization of silicalite-1. The separation factor was as high as 600 for the selective separation of isomers of butanes from hexanes. Huang and Chen (6) studied the effect of addition of polyvinyl alcohol (PVA) as a drying control additive on the microstructure and gas permeation characteristics of supports made from α-alumina and γ-alumina by the slip-casting process. The pore volume and porosity of the support were found to increase with a higher PVA dosage, but the pore size distribution and the tortuosity factor did not undergo any significant change. The pore structure also remained almost invariant with the addition of PVA up to 0.5–10%. Lin and Burggraaf (7) used the chemical vapor deposition technique to modify the pore size of microfiltration and ultrafiltration alumina membranes. Another attempt to modify membranes was reported by Trocha and Koros (8) who successfully plugged very big pores or defects in a membrane by caulking treatment using aqueous colloidal silica. A majority of 2000 Å pores could be closed using 100 to 200 Å colloidal silica sol. Larbot et al. (9) used a bohemite sol to slipcast porous tubes which were sintered by “flash” firing. They avoided an organic binder and used a coating time of about 15 minutes and obtained a molecular weight cutoff of a few hundreds.

In spite of the substantial volume of literature on ceramic membranes, more attention needs to be given to elucidation of the relationship between the conditions of preparation and the ultimate porosity and pore structure of the support and of the permselective layer. In the present work we report how the firing temperature and time influence the pore structure and water permeation characteristics of a membrane support. The effect of the dipping and firing cycles on membrane formation and separation properties of a permselective membrane layer in terms of solvent flux and percentage rejection of solute has been studied. In addition, the potential of Gangetic silt for the preparation of a membrane support as well as the separation performance of an alumina and titania permselective layer formed on this support has been explored.

MATERIALS USED

Commercial grade α-alumina was used without further purification. Chemical analysis of this material is given in Table 1. Gangetic silt was obtained from Falta Firebrick Company, and its chemical analysis is also shown in Table 1. Aluminum tri-isopropylate and tetra-isopropyl ortho-titanate were the organometallic precursors (MERK-Schuchordt, Germany) used for the preparation of sols for coating the support disks. Polyvinyl alcohol of 125,000 and 14,000 dalton molecular weight (S. D. Fine Chem. Ltd.) was used as the binder and drying control additive. Bovine serum albumin of 66,000 dalton molecular weight (Loba Chemie) was used as a standard solute to determine the filtration efficiency of the membranes. Other chemicals used were of analytical reagent quality.

PREPARATION OF SUPPORTED MEMBRANES

Preparation of alumina and titania membranes used in this work involved the following steps:

1. Preparation of α-alumina and clay supports
2. Preparation of stable sols from the organometallic precursors
3. Coating the supports with the sols, followed by firing under controlled conditions

TABLE 1
Chemical Analysis of α-Alumina and Clay (Gangetic silt) Used for Preparation of Supports

Name of the components	Percentage in α-alumina	Percentage in clay
Alumina (Al_2O_3)	97.25	14.15
Silica (SiO_2)	0.85	62.85
Ferric oxide (Fe_2O_3)	0.25	6.05
Calcium oxide (CaO) and		3.51
magnesium oxide (MgO)	0.15	2.89
Alkalies	Traces	—
Loss on ignition	—	10.18

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Preparation of Membrane Supports

α-Alumina (200 g, calcined at 1000°C for 2 h) was mixed with 50–55% of its weight of water and then wet-ground in a ball mill for 2 hours. Four grams of PVA (MW 125,000 dalton) dissolved in 25 mL water was added to the slurry, and milling was continued for 17–18 hours. The material was dried and ground again. About 30 g (−100 mesh) of the material was made into a circular disk (65 mm diameter) in a hydraulic press under 155 Mbar pressure. The green disks were dried to remove any residual moisture at room temperature and then heated at 200°C for 24 hours, and the temperature was increased to 1300–1450°C at a rate of 10°C/min. Firing of the disks was done for at least 2 hours. Clay supports were prepared by an essentially similar procedure. Here, 55–65% of the weight of clay was used to make the slurry, and grinding continued for 6–8 hours. About 30 g (−70 mesh) of the dried material was used to make a green disk at 155 Mbar pressure. The disks were heated for 2 hours at 200°C; then firing was done at 1050–1100°C for 2 hours.

Preparation of the Sols

One mole of aluminum isopropoxide was mixed with 120 mol water and 0.23 mol nitric acid (as a 0.2 M solution), refluxed at 80–90°C for 2 hours, and then evaporated to obtain the desired sol concentration of 0.54–1.4 mol/L. The pH was adjusted to about 3.6. The recipe for the preparation of titania sols was 1 mol tetra-isopropyl ortho-titanate, 76 mol water and 1 mol HNO_3 as a 0.2 M solution. Other steps were as before. The sol concentration was maintained at 0.37–0.945 mol/L and the pH was adjusted to about 2.4. Properties of the sols, including stability, are given in Table 2. Here, stability of a sol is indicated by the period for which it remains intact and can be used for membrane preparation.

Membrane Formation

An ultrathin membrane layer was formed on a support by dip coating, drying, and firing. A support disk was first put in boiling water for some time to expel the air from the pores. The contents were then cooled, and the disk was allowed to surface dry for 2–5 minutes. The disk was dipped in a 2% PVA solution (MW 14,000 dalton) that acted as the drying control additive (10, 11). The sol was poured down the surface of the disk for 5–10 seconds when gelation occurred. This was followed by controlled drying for over 24 hours. Repeated dipping and drying cycles were necessary to get a coating of the desired thickness. The disks were fired in a furnace while maintaining a temperature gradient of 3–4°C/min. The firing temperature for the alumina membranes

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TABLE 2
Properties of Alumina and Titania Sols

Molar ratio of H_2O/alkoxide/HNO_3	Refluxed at 80°C (h)	Evaporated at 90°C (h)	Name of the sol	Final concentration (mol/L)	Sol pH	Stability (months)
100/1/0.15	1.00	—	Alumina	0.635	3.50	10
PVA: 0.277 g						
120/1/0.23	3.50	2.33	Alumina	1.407	3.80	4
120/1/0.23	2.75	1.25	Alumina	0.563	3.70	6
120/1/0.23	2.75	1.0	Alumina	0.540	3.67	7
120/1/0.23	2.00	0.5	Alumina	0.360	3.40	6
200/1/0.20	1.5	—	Titania	—	—	No sol
PVA: 0.1 g						
73/1/0.32	2.0	1.0	Titania	0.945	2.0	3
73/1/0.3	2.0	0.75	Titania	0.472	1.9	12
76/1/0.25	2.0	0.08	Titania	0.370	2.4	13

was in the 600–800°C range and that of the titania membranes was 450–550°C. It was found that 2–4 dips and 2 firings were sufficient to form membranes of 3–5 μm thickness.

Data on the conditions for the preparation of the supports and membranes are given in Tables 3a and 3b, respectively.

TABLE 3A
Identification of Supports

		Firing/sintering			
Support	Casting pressure (Mbar)	Temperature (°C)	Time (h)	Thickness (mm)	Porosity (vol%)
A1	155	1400	7	4.68	46
A2	155	1440	7	5.00	39
A3	155	1300	19	3.14	34
		1440	1.5		
A4	155	1300	19	3.23	33
		1420	3		
C1	155	1050	14	3.60	26
C2	155	1050	2.5	4.00	32
C3	155	1100	2.0	4.10	26
C4	155	1100	1.75	4.00	29
C5	155	1100	2.0	4.35	26

TABLE 3B
Identification of Membranes

Membrane	Support	Sol concentration (mol/L)	pH	No. of dippings	No. of firings	Firing temperature (°C)
A2T2	A1	0.54	3.67	2	2	750
		0.37	1.23	2		470
T2T2	A2	0.37	1.23	4	2	480
A2A1	A3	0.54	3.67	3	2	750
A2A2	A4	0.54	3.67	4	2	750
CT3	C1	0.37	2.40	6	3	480
CT4	C1	0.37	2.40	8	4	480
CA3	C5	0.54	3.67	6	3	740

CHARACTERIZATION OF SUPPORT AND MEMBRANES

Characterization of the membrane support was done by determining the porosity, pore size distribution, and average pore diameter using a mercury porosimeter (Autopore 11, 9220) at a maximum mercury penetration pressure of 60,000 psia. Figures 1 and 2 show the pore size distribution of alumina and clay supports sintered at different sets of conditions. The pore diameter of a

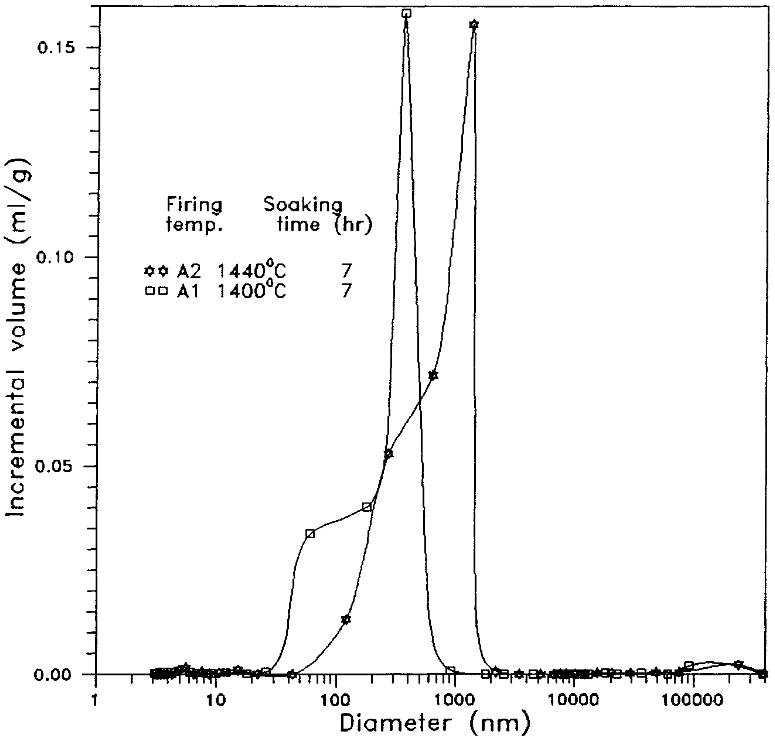

FIG. 1 Pore size distribution of alumina support. Effect of sintering temperature.

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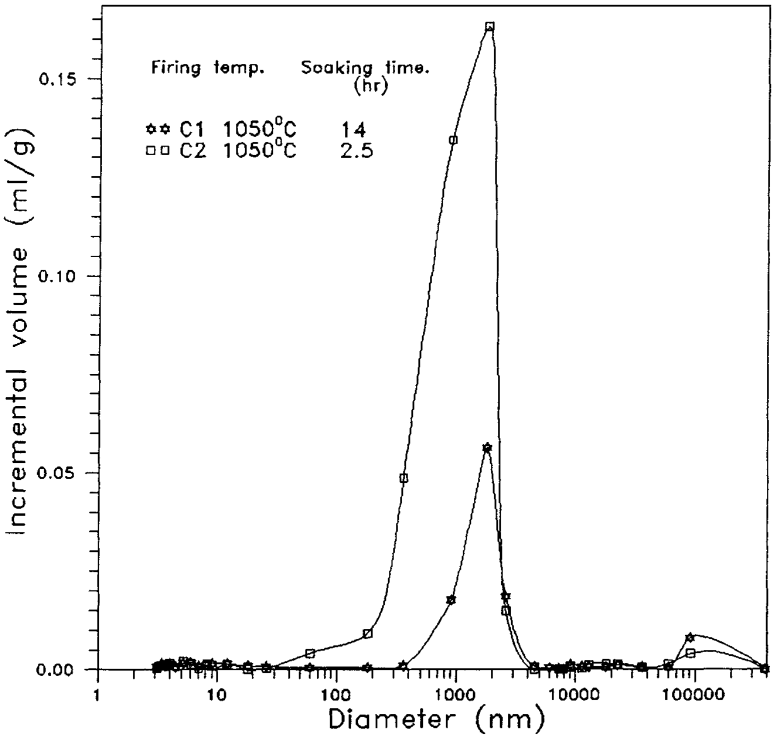

FIG. 2 Pore size distribution of clay support sintered at 1050°C for different soaking times.

typical alumina support (sintered at 1400°C) lies in the 20–1000 nm range (Fig. 1), but the range of the pore diameter increased to 40–2500 nm when the support was sintered at 1440°C for the same duration. A similar effect of firing temperature was reported by Leenaars et al. (12). They observed an increase in the average pore diameter from 3 to 180 nm for titania membranes when the sintering temperature was changed from 400 to 1170°C.

Pore size distribution of a clay support sintered at 1050°C is presented in Fig. 2, as is also the effect of firing time on pore size distribution. The range of pore diameters increased from 30–4500 to 300–4500 nm when the duration of firing was increased by about 5 times. Porosimetry studies indicate that with an increase of temperature and firing time, pore diameter increases but porosity decreases. This is because adjacent pores tend to fuse together to form bigger pores at an elevated firing temperature. Some of the pores may even disappear during sintering, and this results in shrinkage of the disk.

The membrane morphology, pore diameter, and determination of membrane thickness were done by SEM studies (S415A, Etachi, Tokyo) of supported membranes. A picture of the top surface of the membrane (A2A2) showing the structures of the pores, which are mostly elongated, is given as Fig. 3, and that of the fractured section of the membrane is given as Fig. 4 which shows the pores of the supported membrane.

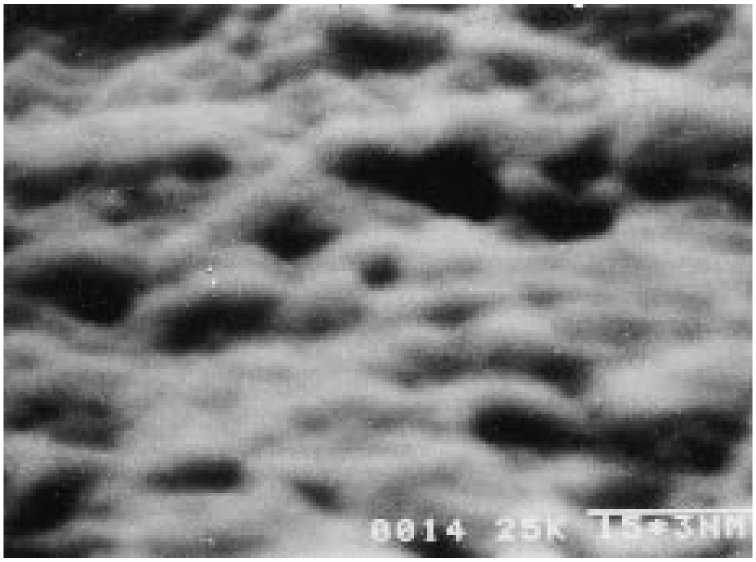

FIG. 3 Scanning electron micrograph of the upper surface of an alumia-on-alumina membrane (A2A2).

EXPERIMENTAL

Water permeability and ultrafiltration studies were carried out in a flanged, two-compartment, 5 cm i.d. pressure cell. The membrane rested on a perforated disk separating the compartments, and a neoprene “O” ring was compressed against the membrane to prevent leakage. Air pressure was applied to the upper compartment during a run. The effective permeation area was 13.2 cm^2.

FIG. 4 Scanning electron micrograph of the fractured section of the A2A2 membrane.

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A motorized agitator was fitted to the cell through a stuffing box in order to reduce the concentration polarization at the membrane surface during filtration. During an experiment a measured amount of water or solution was taken in the upper compartment of the cell connected to the air tank of the compressor. The compressor was set at the desired pressure. The permeate was collected from the bottom compartment of the cell over different time periods. The agitator was maintained at a desired rpm during filtering of the albumin solutions so that concentration polarization could be reduced but no denaturation of protein would occur. Samples of albumin solution were collected at the beginning and at the end of an experiment. They were analyzed for their solute content at 300 nm using a UV-Vis spectrophotometer (Shimadzu TCC-240A).

RESULTS AND DISCUSSION

In order to investigate the functional properties of the membranes, a series of runs was made on the permeation flux of pure water and of solutions of bovine serum albumin at different solute concentrations and transmembrane pressure drops. The results of such measurements are described below.

Water Permeability of Support Disks

Porosity and thickness of the disks as well as the firing time and temperature are likely to have a significant influence on water flux through the uncoated disks. Typical results on water flux for a few alumina supports are given in Fig. 5. It shows that at similar firing temperatures, a lower firing time causes a higher porosity, and a higher porosity predictably allows a higher water flux. Figure 5 for alumina supports also shows that a reduction of porosity from 46% (support A1; 7 h at 1400°C) to 39% (support A2; 7 h at 1440°C) decreased the water flux by a factor of about 2. The pore size distributions of these supports (Fig. 1) shows that the average pore diameter of A2 is about 500 nm compared to about 250 nm of A1. The reduced water flux (despite the bigger average pore diameter) for support A2 may be due to the fact that many of the smaller pores fuse together to generate a network of bigger pores at a higher firing temperature, but the fraction of effective pores decreases (9). Fusion of pores associated with shrinkage may give rise to dead-end pores. Such pores contribute to the pore-size distribution plot but not to the water flux. The linear dependence of water flux on pressure drop across a support indicates Poiseuille flow through the pores. The flow path of the liquid through a support is complex and depends upon the network structure of the pores and their size distribution. However, an analysis of the flux data of Fig. 5 indicates that q/ϵ^4 (where q is the water flux and ϵ

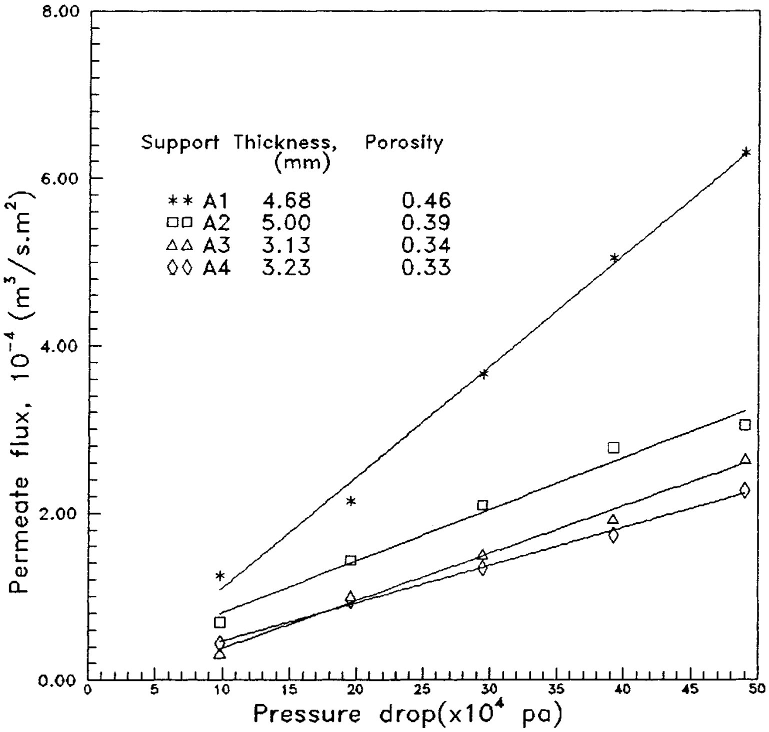

FIG. 5 Water flux through alumina supports of various porosities and thicknesses.

is the porosity) remains constant to within about 7% for a unit thickness of the support.

The water flux through a 4-mm thick clay support was about 10 times higher than that of an alumina support. This was because there is an order of magnitude larger pore size in the clay support. The water flux was also found to be linear in the transmembrane pressure drop. However, unlike alumina supports, a flux decline with time was observed for clay supports. An asymptotic water flux was reached after several hours (Fig. 6). This can be explained by the "hydration effect" of the pores. The pores swelled when hydrated, and therefore the flux decreased. The phenomenon was confirmed by repeating the permeability test after drying the support properly when the original flux profile was restored (Fig. 6). In order to test the hydration effect, water sorption of the clay support was measured. A piece of dry support was put in boiling water for 15 minutes, cooled, and allowed to stand for an hour. The piece was then surface dried and weighed. Water sorption was 0.1681 g per g of dry support. This was about 30% higher than the porosity of the same support (0.1267 cm^3/g) obtained from mercury porosimetry. Thus a piece of clay support took up 30% more water than its pore volume, and this excess water accounts for the hydration. For a piece of alumina, however, the porosity as obtained by

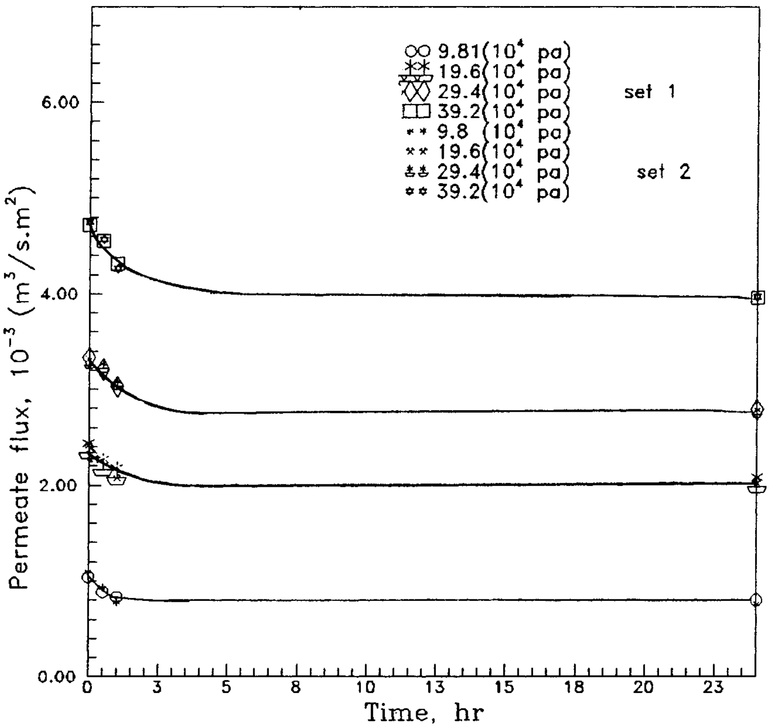

FIG. 6 Variation of water permeability of clay support with time (thickness = 4.0 mm, porosity = 29%). Set 1: Data for fresh support. Set 2: Data for support after drying. The pressure drops are given in parentheses.

mercury porosimetry (0.2426 cm^3/g) was almost the same as the water sorption (0.2392 g water per g support).

ULTRAFILTRATION OF ALBUMIN SOLUTION

The ultrafiltration performance of the membranes was tested by separation of bovine serum albumin (BSA) of 66,000 dalton molecular weight from aqueous solutions of different concentrations with or without stirring. The filtration rate of BSA solution was an order of magnitude lower than that of pure water flux through the membranes. Further, pure water flux through a support was nearly equal to that through the corresponding supported membrane. This indicates that the hydraulic resistance of the ultrathin membrane layer was rather small compared to the resistance of the support.

The concentrations of BSA in the permeate and retentate were determined from the respective absorbances measured by a spectrophotometer. The rejection coefficient of a solute is expressed as follows:

$$\text{Percent rejection} = 100(1 - [C_{\text{permeate}}/C_{\text{feed}}])$$

Ultrafiltration of Albumin Solution through Alumina-Supported Membranes

Filtration of BSA solutions was done using alumina supports at different transmembrane pressure drops. The decline of flux with time was more rapid at a higher pressure drop. The rejection of BSA from a 2% solution by using a support as the barrier was 5 and 10% for pressure differentials of 98 and 294 kPa, respectively. The flux decline was due to partial blockage of the pores of the support.

Figure 7 shows the effect of stirring on the filtration of a 2% albumin solution through the alumina membrane marked A2A1 (three dips, two firings; sol concentration = 0.56 mol/L, pH 3.7). It was observed that the rejection coefficient increased from 0.73 to 0.77 when the contents of the cell were kept stirred. The permeate flux was also enhanced by about 20 to 30% because of a reduction of concentration polarization by stirring. No quantitative estimation of the effect of concentration polarization was done.

Water permeability and filtration rate of 2% albumin solution were measured for the membranes A2A2 (four dips, two firings), prepared using the same sol but on two different alumina supports (A3 and A4). A better per-

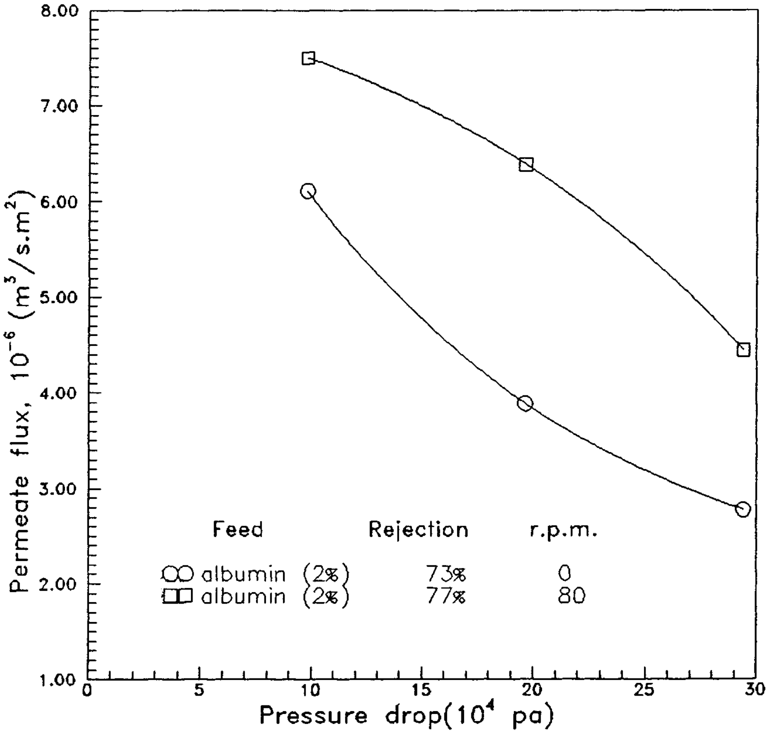

FIG. 7 Filtration of 2% albumin solution through alumina-on-alumina membrane. Effect of agitation.

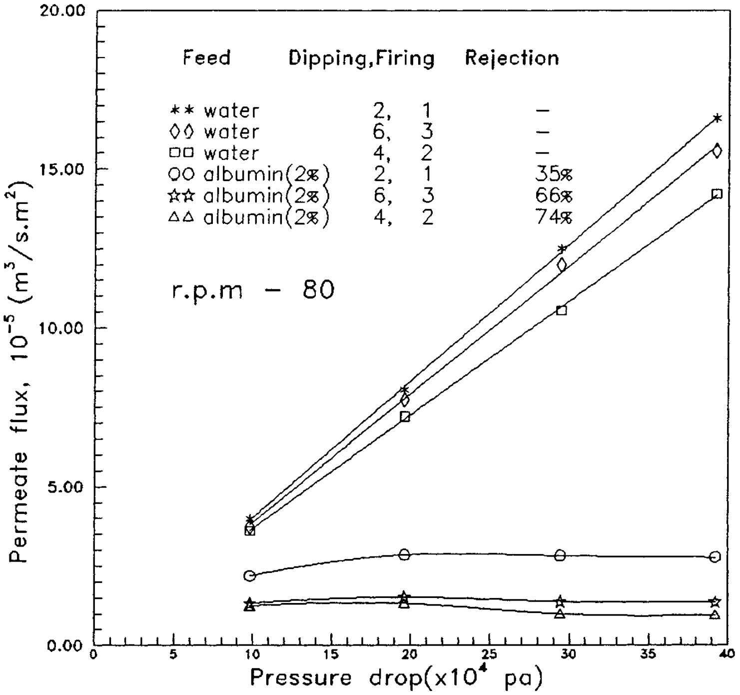

FIG. 8 Water permeability and filtration of 2% albumin solution through titania-on-alumina membrane. Effect of firing cycle.

formance was achieved with a rejection coefficient of 0.79 and 0.80 compared to the previous membrane (A2A1) described above. The results establish the reproducibility of membrane performance. It was also indicated (Fig. 8) that a single dipping and firing cycle did not produce a good membrane, and the membranes formed remained imperfect and nonuniform. A further coat of sol removed these imperfections although it increased the membrane thickness.

Although repeated dipping and firing cycles were found to be necessary for making good membranes [in fact, some researchers repeated the cycles three times with one to four dippings in each cycle; e.g., Peterson and Anderson (13)], our results are at variance with this. In order to find out how many dipping and firing cycles were necessary, we coated the alumina support A2 thrice with intermediate firing using a 0.37 mol/L titania sol at a pH of 1.23. It showed that the rejection increased from 35 to 74% after four dippings and two firings (Fig. 8). A further double dip followed by firing, in fact, caused a lowering of the rejection to 66%. In order to establish this result more decisively, four additional dips and two firings were done. The result was a drastically low rejection of BSA. Serious defects grew after such a large number of dippings and firings. A SEM picture of this membrane also showed scattered cracks on the surface. This was because of the strain developed in successive layers of coating caused by repeated firings.

Figure 9 shows the effect of stirring on the filtration of a 2% albumin solution and the performance of titania on an alumina membrane. A 2% albumin solution was filtered through a twice fired (6 dips) titania membrane (sol concentration = 0.172 mol/L; pH 2.4) with or without stirring. A rejection of 66% was achieved under the nonagitated condition; stirring enhanced the rejection to 75%. Flux increased with an increase of transmembrane pressure drop as expected, but it began to decrease when a barrier of solute particles formed on the membrane surface after some time.

Filtration performance of a 4% albumin solution was also determined for the alumina–alumina (A2A2) and alumina–titania (A2T2) composite membranes. It was found that rejection of albumin by the A2A2 membrane was about 53%. Both rejection percent and filtration rate were less than those of a 2% albumin solution. An increase of the protein in the permeate with an increase of the feed concentration was also reported by Hvid et al. (14).

It is relevant to compare the performance of the above membranes with those of other membranes as reported in the literature. Peterson et al. (2) reported 95% rejection of polyethylene glycol (MW = 100,000 dalton) for an Alcoa alumina membrane. A similar percent rejection for BSA (MW =

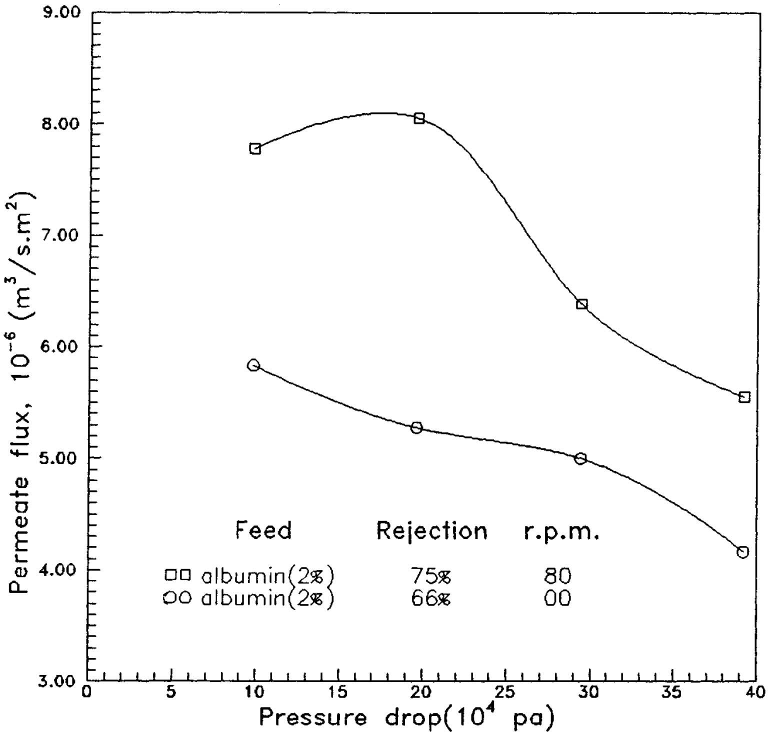

FIG. 9 Filtration of 2% albumin solution through titania-on-alumina membrane. Effect of stirring.

67,000 dalton) by an alumina membrane was reported by Itaya et al. (15). Langer and Schnable (16) obtained only 65% rejection of BSA (MW = 69,000 dalton) when using a porous glass membrane of 15 nm average pore size. The rejection percent for BSA achieved in this work is less than that reported by Peterson and Itaya but considerably more than that achieved by Langer and Schnabel (16).

From a comparison of the rejection achieved by our membranes with the rejection data on BSA (MW = 69,000 dalton) for membranes of different pore sizes, together with SEM studies, we estimate that the average pore size of the membranes used in this study varied between 12 to 17 nm.

Ultrafiltration of Albumin Solution through Clay-Supported Membranes

Formation of crack-free clay-supported membranes is a difficult process. The presence of a small fraction of bigger pores may be responsible for defects in these membranes. The filtration performance of clay-supported membranes was not as good as that of the alumina-supported membranes. A moderate degree of rejection could be achieved using clay-supported titania membranes.

Six times titania-coated (sol concentration = 0.37 mol/L) clay support was fired thrice at 500°C. This membrane (CT3) rejected about 51% (Fig. 10) sol-

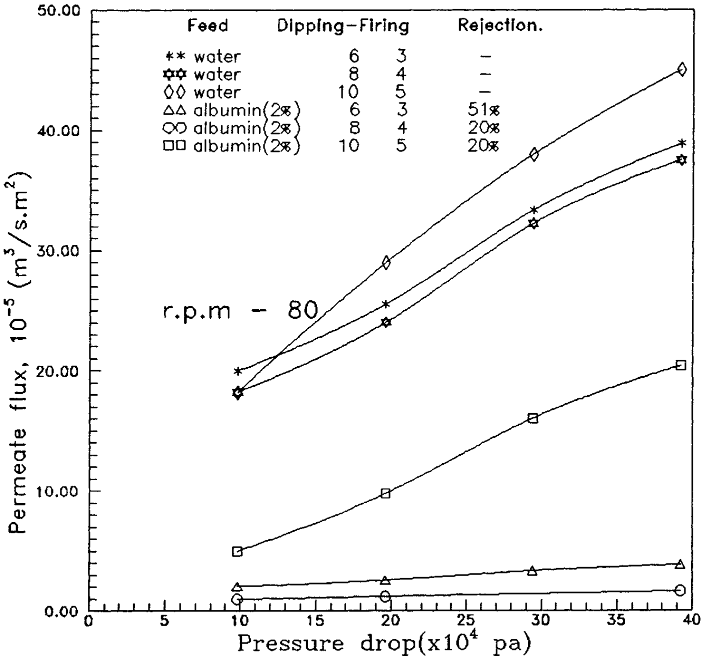

FIG. 10 Water permeability and filtration of 2% albumin solution through clay-supported titania membrane. Effect of dipping.

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ute from a 2% albumin solution. If the supported membrane was further doubly coated with the same titania sol and then fired, the rejection reduced from 51 to 29%. If the coating, drying, and firing were repeated 5 times, the rejection percentage did not increase further but the flux increased remarkably due to cracking of the membrane. This result also indicates that the required numbers of dipping and firing are higher for clay-supported membranes than for alumina-supported membranes.

CONCLUSIONS

Commercial α-alumina and Gangetic silt have been used for the preparation of alumina and clay supports. Alumina supports were sintered at 1300–1450°C and clay supports at 1050–1100°C for varying times. Firing temperature and time have significant effects on porosity and pore size distribution of the supports. A support fired at a higher temperature underwent compaction, the average pore size became larger, and the pore size distribution widened (porosity of supports decreased at higher firing temperatures but the average pore size increased). The supports themselves could marginally separate albumin of 66,000 dalton molecular weight. Appropriate preparation conditions of alumina and titania sols from the corresponding alkoxides were identified so that the stability of the sols could be greatly improved. The number of coating and firing cycles necessary to make defect-free membranes, as indicated by the SEM pictures and filtration performance, were also established. Water permeability studies of the membranes indicated negligible hydraulic resistance of the thin permselective layer. The water permeability of a clay membrane decreased with time because of hydration but reached an asymptotic value after a few hours. The flux increased with increasing transmembrane pressure drop during filtration but began to fall when a solute barrier formed on the surface. Stirring the liquid increased the flux by about 20–30%. Both rejection and filtration rate decreased with an increase in the concentration of the feed solution. All the alumina-supported membranes had good rejection characteristics for albumin solutions, but only a moderate degree of rejection could be achieved by the clay-supported membranes in their present form.

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