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Polysulfone membranes. IV. Performance evaluation of Radel A/PVP membranes*

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Abstract

Nuclear magnetic resonance (NMR) spectroscopic measurements were used to show that Radel A100 is a copolymer containing polyethersulfone and polyetherethersulfone repeat units. Membranes were cast from solutions of Radel A and polyvinylpyrrolidone (PVP) polymers dissolved in 1-methyl-2-pyrrolidinone (NMP). The variation in membrane pore size is related to the casting solution composition and viscosity. The performance of Radel A/PVP membranes is compared to those of commercially available polysulfone membranes.

Keywords: ultrafiltration; pore radius; Radel A100; polyethersulfone membranes; polysulfone membranes

Introduction

Radel A100 polyethersulfone [1] (Radel A) is an engineering plastic manufactured by Amoco Performance Products. Neither the complete chemical structure of this polymer nor its membrane properties have been reported in the literature. The purpose of this work is to determine whether Radel A membranes have similar permeation and separation characteristics to those made from Victrex polyethersulfone (PES). This issue is pertinent since ICI recently ceased production of Victrex PES, even though this material is widely used to make commercial ultrafiltration membranes.

In this work, the structure and composition

of this polymer was determined by nuclear magnetic resonance (NMR) spectroscopy. Membranes were prepared from casting solutions of various concentrations of Radel A and polyvinylpyrrolidone (PVP) in 1-methyl-2-pyrrolidinone (NMP). Membranes were compared based on two structural parameters: the average pore radius and the pore density to pore length ratio. These parameters were obtained by using a steric transport model to describe the membrane sieving experiments.

Theory

A membrane is considered to have a microporous surface. The pore morphology can be represented by a series of parallel and cylindrical pores. Fluid movement through the membranes can be described by standard continuum fluid mechanics. Solute separation arises

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from steric and hydrodynamic interactions between the solute and the pore wall. A complete derivation of this transport model is reported elsewhere [2-4].

Solute separation, f , is a function of the pore Peclet number so that,

$$f = 1 - \frac{\chi}{1 - e^{-Pe(1-\chi)}} \quad (1)$$

The pore Peclet number, Pe , is the ratio of the convective and diffusive transport of the solute through the pore. This dimensionless parameter is defined as

$$Pe = \frac{\chi}{\xi D_{\infty}} \left(\frac{r_p^2 \Delta P}{8\eta} \right) \quad (2)$$

where η is the viscosity of water, ΔP is the pressure drop across the membrane and r_p is the average pore radius; χ is a global steric parameter associated with the restricted convective transport within the pore; ξ is the ratio of restricted diffusivity of the solute within the pore to the bulk diffusivity of the solute in solution (D_{∞}); χ and ξ are available as a function of the solute radius and average pore radius [2].

An estimate of the porosity and the resistance to flow can be obtained from the pure water permeation rate [5]. The Hagen-Poiseuille equation relates the volumetric flow rate (Q) to the pore size, number of pores (n) and pore length (Δx), so that,

$$Q = \frac{n\pi\Delta P r_p^4}{8\eta\Delta x}, \quad (3)$$

where Δx represents the overall hydraulic resistance to water transport. The ratio of the number of pores to the pore length per unit area (A) can be obtained from a pure water permeation rate by re-arranging the above equation as,

$$\frac{n}{\Delta x A} = \frac{Q 8\eta}{\pi \Delta P r_p^4 A} \quad (4)$$

where r_p has been determined from solute sieving experiments.

Experimental

Materials

Radel A100 polyethersulfone was obtained from Amoco Performance Products Inc. and was dried before using. The solvent for making the membrane casting solution was reagent grade NMP (Anachemica) and was used as received. Polyvinylpyrrolidone (PVP 10,000 M_w) was obtained from Sigma and dried in a vacuum oven overnight before use. Poly(ethylene glycol)s (PEG) with nominal molecular weights ranging from 600 Da to 35,000 Da were purchased from Fluka and were used as solutes in the sieving experiments. Solvents for solubility measurements were obtained from Anachemica and Aldrich.

Material characterization

NMR

A sample of Radel A was dried at 130°C overnight, then dissolved in DMSO- d_6 containing an internal tetramethylsilane reference. 1H -NMR and ^{13}C -NMR spectra were recorded on a Bruker AM-400 spectrometer at room temperature. ^{13}C -NMR spectra were recorded with 1H noise decoupling. Chemical shifts (δ) are expressed in parts per million (ppm) and coupling constants (J) are in Hertz.

Solubility

Radel A (5 wt.%) in various solvents were left to stand at room temperature for 48 hr. After this period, the degree of solubility of the polymer in the solvent was determined visually.

Membrane preparation

Casting solutions were prepared from Radel A/PVP mixtures dissolved in NMP. Solution viscosities were measured at 25°C using a Haake (M500) viscometer. Casting solutions were prepared for all combinations of Radel A

at 15, 20 and 25 wt.% and PVP at 0, 5, 10, 15, 20 and 25 wt.%. Membranes were prepared by casting a 254 μm (0.010 in) film on a spun bound polyester backing (Hollitex 3296) and gelling in reverse osmosis treated water at 4°C. Residual NMP was leached from the membranes by soaking them in water replaced daily over a three day period.

Membrane characterization

Membrane testing procedures for determining solute separation curves were the same as for those previously reported [5]. Ultrafiltration experiments were performed in crossflow test cells with an effective membrane surface area of $14.5 \times 10^{-4} \text{ m}^2$. Feed flowrate was 3 L/min providing a crossflow velocity of 0.8 m/sec. All test runs were performed at an operating pressure of 344 kPa (50 psig) or 69 kPa (10 psig) depending on the pore radius of the membrane.

A PEG feed concentration of 200 mg/L was used for the solute sieving experiments. This low concentration reduced the possibility of concentration polarization at the membrane surface. Feed and permeate concentrations were determined using a Shimadzu 5000 Total Organic Carbon analyzer.

Scanning electron microscopy (SEM) pictures were obtained for some of the membranes. Cross-sections of the membranes were obtained from freeze fractured dried membranes. The membranes were gold-sputtered coated to provide electrical conductivity. SEMs were taken with a JOEL JSM-84 electron microscope operating at 10 kV.

Results and discussion

¹H-NMR

Radel A100 polyethersulfone (Fig. 1) is described as a modified polyethersulfone (PES) containing low levels of polyetherethersulfone

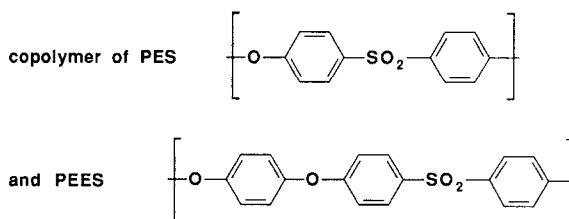


Fig. 1. Structural repeat units of Radel A polyethersulfone.

(PEES) units. It is produced from 4,4'-dichlorodiphenylsulfone and 4,4'-dihydroxydiphenylsulfone with a low level of a second proprietary diphenol [6].

The proton NMR spectrum (Fig. 2) indicates that Radel A is a wholly aromatic polymer. The two main resonance signals are coupled doublets at $\delta=8.01$ (H_d) and $\delta=7.28$ (H_e , $J \sim 8.7$), arising from polyethersulfone (PES) repeat units in the copolymer chain. These resonances are practically the same as those of Victrex PES. Three minor resonances are a singlet at $\delta=7.23$ (H_a), a doublet at $\delta=7.16$ (H_b) and a doublet at $\delta=7.97$ (H_c). These are consistent with a polyetherethersulfone (PEES) repeat unit. The singlet arises from the four equivalent protons on the phenyl ring *para* substituted with ether linkages. Protons *ortho* to the ether linkage on the phenylsulfone portion of PEES give rise to the upfield doublet ($\delta=7.16$) and those *ortho* to sulfone are deshielded ($\delta=7.97$) and are close to the PES *ortho*-sulfone proton doublet. These doublets were shown to be coupled by resonance decoupling experiments.

Condensation polymerization of 4,4'-dichlorodiphenylsulfone with an alkali metal salt of 4,4'-dihydroxydiphenylsulfone produces a polymer with PES repeat units, or more accurately, polyethersulfone-ethersulfone (PESES) repeat units. Polymerization of 4,4'-dichlorodiphenylsulfone with the salt of the proprietary diphenol yields a polymer with PEES repeat units (Fig. 3). Logically, this diphenol is hydroquinone. A representative

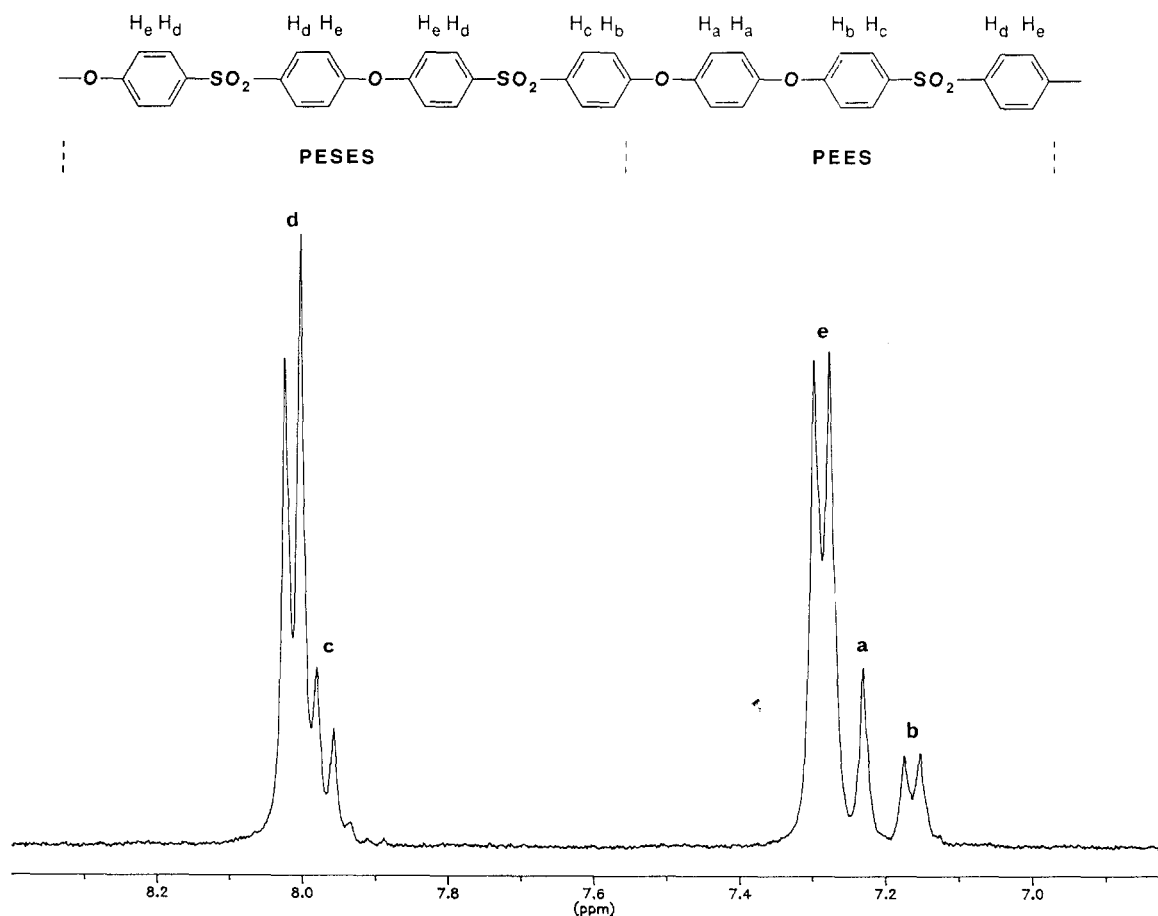


Fig. 2. ^1H -NMR spectrum of Radel A polyethersulfone.

PESES-PEES segment produced from these monomers is shown in Fig. 3. The ratio of PEES to PES in the copolymer can be calculated by integrating the appropriate signals corresponding to each unit. The two types of ortho-sulfone protons H_c and H_d have closely similar chemical shifts that do not allow accurate individual integration. The two small upfield signals H_a and H_b each represent four protons of a PEES repeat unit. Proton signal H_b is more convenient to measure because of its chemical shift separation from other signals. Therefore, the ratio of PEES to PES in Radel A can be represented by integration of the following signals:

$$\frac{\text{PEES}}{\text{PES}} = \frac{[\text{H}_b]}{[\text{H}_c + \text{H}_d] - \text{H}_b}$$

A calculation of this ratio gave a PEES to PES ratio of approximately 0.18:1. However, this value is slightly below the range of 20-30% PEES units [6] recently disclosed by Amoco Performance Products, Inc.

^{13}C -NMR

Figure 4 shows the ^{13}C -NMR spectrum of Radel A polyethersulfone with the resonance assignments. A ^{13}C -NMR spectrum of Victrex PES (not shown) was used to assign the reso-

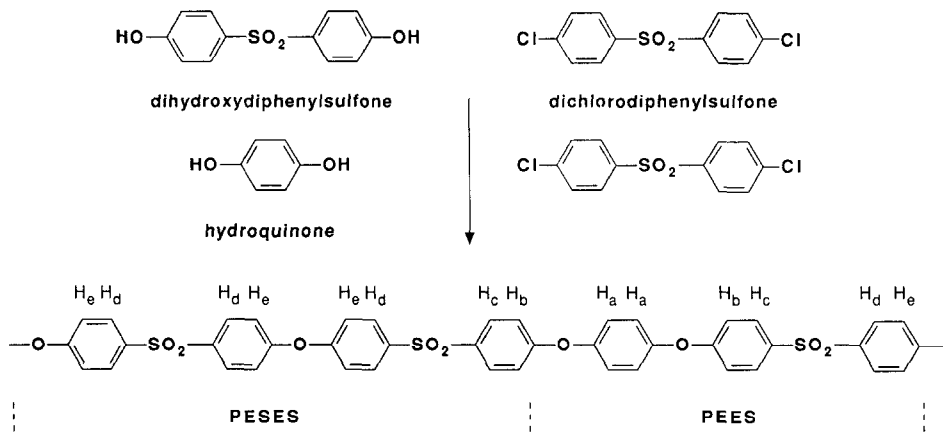


Fig. 3. Synthetic scheme and representative PESES-PEES segment of Radel A polyethersulfone.

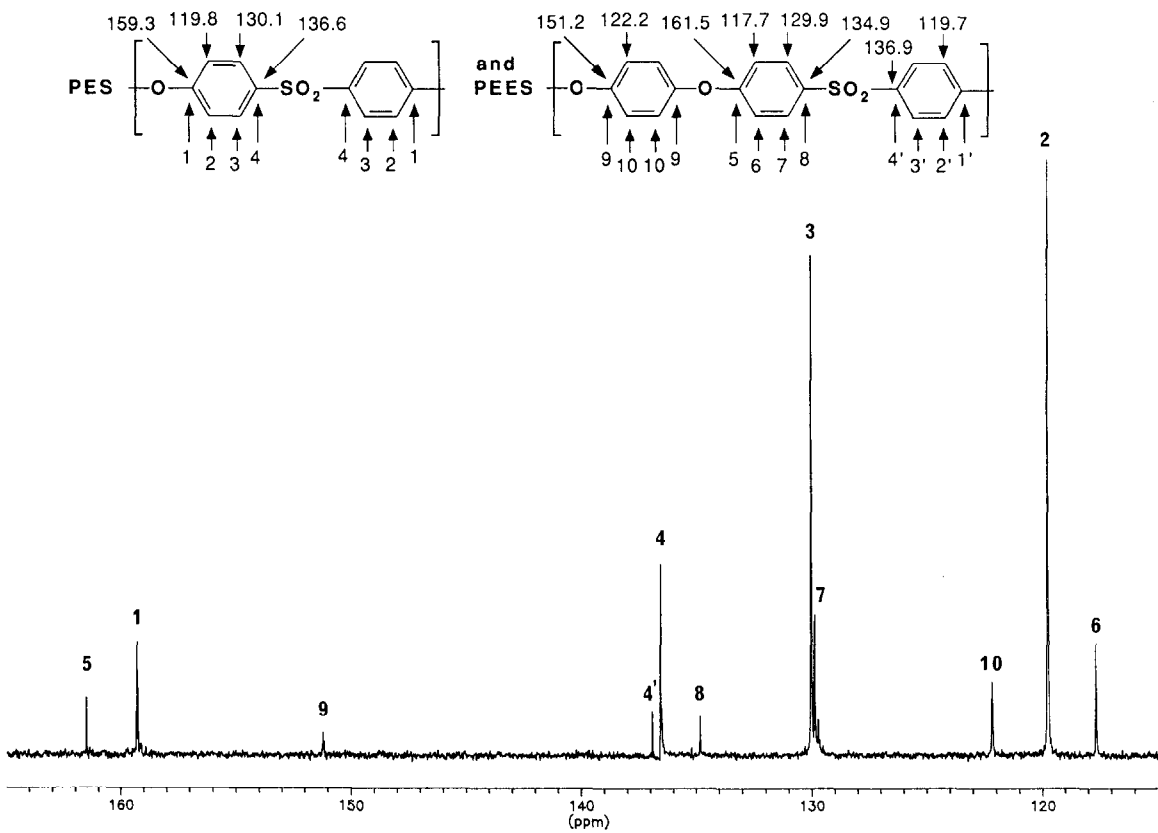


Fig. 4. ¹³C-NMR spectrum of Radel A polyethersulfone.

TABLE 1

Elemental analysis of commercial polysulfones

Polysulfone	Elemental (%)	Theoretical			Experimental		
		C (%)	H (%)	S (%)	C (%)	H (%)	S (%)
Victrex PES 200P	C ₁₂ H ₉ SO ₃	62.06	3.47	13.80	62.36	3.36	12.51
PEES segment	C ₁₈ H ₁₂ SO ₄	66.66	3.73	9.88	-	-	-
Radel-A100 ^a	-	62.75	3.51	13.21	62.51	3.54	12.26
Radel-R5000	C ₂₄ H ₁₆ SO ₄	71.99	4.03	8.01	71.67	3.99	8.03
Udel P1835	C ₂₇ H ₂₂ SO ₄	73.28	5.01	7.24	73.18	4.99	7.26

^aBased on PEES/PES ratio of 0.18:1.

nances of the polyethersulfone repeat unit. The Victrex PES spectrum had four resonance signals (C-1, $\delta=159.3$; C-2, $\delta=119.8$; C-3, $\delta=130.1$; C-4 $\delta=136.6$) that corresponded exactly with the PES segments of Radel A. The chemical shift assignments were made by comparing shifts calculated using additivity rules for substituted benzenes [7]. Proton-carbon one bond connectivities were also obtained from two dimensional (2D) spectra. The chemical shifts and assignments for PES are in close agreement with the values given for PES macromers [8]. A PEES segment has a high probability of being connected to a PESES segment because of the relative ratios. Therefore, there will be some small differences between the chemical shifts for Radel A PEES segments and for a polymer composed entirely of PEES repeat units. Chemical shifts for PES segments (C-1-C4) adjacent to PEES should be closely similar to C-5-C-8 because of equivalence about the hydroquinone portion. Two carbon resonances (C-9, $\delta=151.2$, C-10 $\delta=122.2$) are assigned to the hydroquinone portion of the PEES segment. C-6, C-7 and C-10 are assigned on the basis of 2D spectra. The chemical shifts of the ring adjacent to the hydroquinone portion were significantly shifted from those of PES, with the exception of C-7. Only one chemical shift resonance, C-4, was shifted from PES in the

third ring. C-4 is assigned to the resonance at $\delta=136.9$, being closest to the chemical shift of the equivalent resonance in PES and C-8 is assigned to the resonance at $\delta=134.9$. The statistically low probability of two PEES segments being adjacent to one another is believed to give rise to minor resonances occurring at $\delta=159.3$, 135.2 and 129.7.

Elemental analysis

Elemental analyses for Radel A and other commercial polysulfones were obtained and summarized in Table 1. The theoretical values of C, H and S composition for Radel A were obtained by applying the PEES to PES ratio obtained from NMR. The experimental values agreed well with the theoretical compositions except for sulfur in Victrex PES and Radel A. The presence of PEES in Radel A is supported by the higher percentage of carbon present than in Victrex.

Bulk and solution properties of Radel A

The physical and chemical properties of Radel A compared to other aromatic polysulfones have been reported by Harris and Johnson [9]. The material properties of Radel A are comparable to Victrex polyethersulfone, which is

TABLE 2

Solubility of polysulfones in various solvents (A: soluble; B: soluble/cloudy; C: partly soluble; D: swells; E: slightly swells; F: softens; G: insoluble, p: slight precipitate)

Solvent	Udel	Victrex	Radel A
Benzonitrile	A	A	A
Tetramethylurea	A	A	A
N-Methyl-pyrrolidinone	A	A	A
γ -Butyrolactone	B	A	A
Dichloromethane	B	A	A
Dimethylacetamide	B	A	A
Dimethylformamide	B	A	A
Dimethylsulfoxide	C	A	A
Chloroform	B	B	BC
Cyclohexanone	B	CD	CD
Tetramethylenesulfone	C	D	CE
Bis(methoxyethyl)ether	B	A	D
Pyridine	B	D	D
Dioxane	B	F	D
Tetrahydrofuran	B	F	D
Ethyleneglycoldimethylether	C	C	D
Acetone	D	D	D
Methylethylketone	D	D	D
2(Methoxyethoxy)ethanol	F	Ap	D
Chlorobenzene	A	G	E
Ethylacetate	E	G	E
2-Methoxyethanol	F	G	E
Toluene	D	G	G
1,1,1-Trichloroethane	E	G	G
Acetonitrile	F	G	G
Ethyleneglycol	G	G	G
Hexane	G	G	G
Methanol	G	G	G
Water	G	G	G

not unexpected considering the structural similarities between the two polymers.

The solubility of Radel A was evaluated in various solvents. Table 2 summarises the solvents that can swell or dissolve this polymer. The solubility region of Radel A is comparable to that of Udel polysulfone and Victrex PES. The chemical properties of a polymer can be quantified using solubility parameters [10]. Based on this list of solvents, the total Hansen's solubility parameter for Radel A is calculated to be $22.2 \text{ MPa}^{1/2}$, assuming a spherical

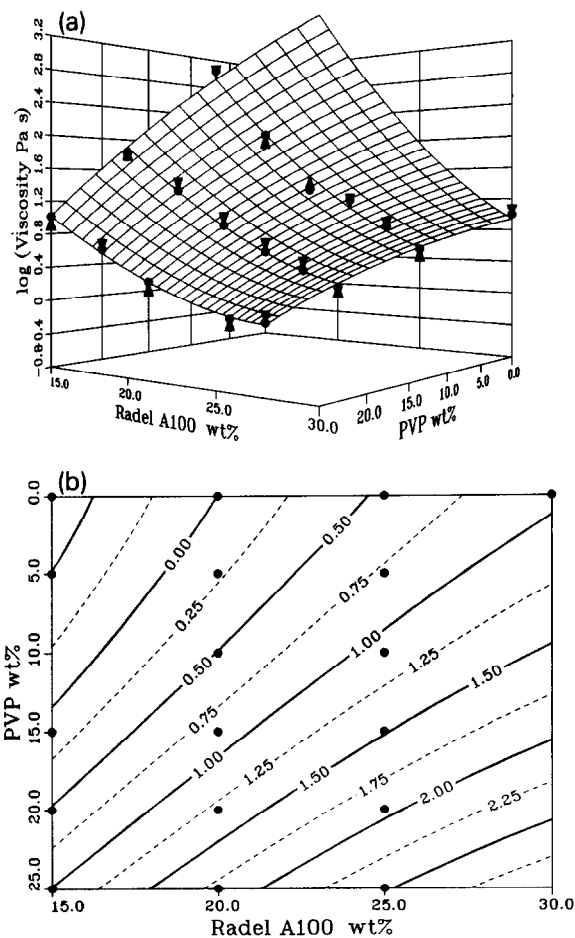


Fig. 5. (a) Logarithm of Radel A/PVP casting solution viscosity as a function of composition. Surface obtained from a polynomial fitted to the experimental data (\bullet) using sum of squares of residuals fitting. (b) Contour plot representation of Fig. 5(a).

solubility envelope. This value is comparable to the solubility parameter for Victrex PES calculated from group contributions. The general rule for the use of solubility parameters suggests good solvents have solubility parameters similar to that of the polymer. A total Hansen's solubility parameter of $22.9 \text{ MPa}^{1/2}$ for NMP is close to the center of the solubility envelope for Radel A. Therefore, NMP can be considered to be a good solvent for Radel A.

The importance of viscosity of the casting

solution for membrane formation has been reported extensively for many polymer solvent systems (e.g. [11,12]). The mixing and processibility of the casting solution is affected by its viscosity.

The addition of PVP to a polysulfone casting solution increases its viscosity. The contour plot, Fig. 5(b), shows the logarithm of the viscosity as a function of Radel A and PVP concentration. The viscosity of the Radel A in NMP reaches only 5.5 Pa-sec for a 50 wt.% casting solution. The polymer solubility limit in NMP at room temperature was reached before higher viscosities could be attained. The addition of PVP had a profound effect on the viscosity, increasing it up to 680 Pa-sec for a 25

wt.% Radel A/30 wt.% PVP solution. Such a high viscosity results in mixing and handling difficulties for these casting solutions. Small values of the solution viscosity at 15% Radel A imposed a low operating limit for casting flat sheet membranes. Below this limit, the casting solution can penetrate the backing material used in this work.

Membrane characteristics

Figure 6 shows SEMs of Radel A/PVP membranes for different solution compositions. Finger-like structures commonly found beneath the skin layer of ultrafiltration membranes are seen. The appearance of these mac-

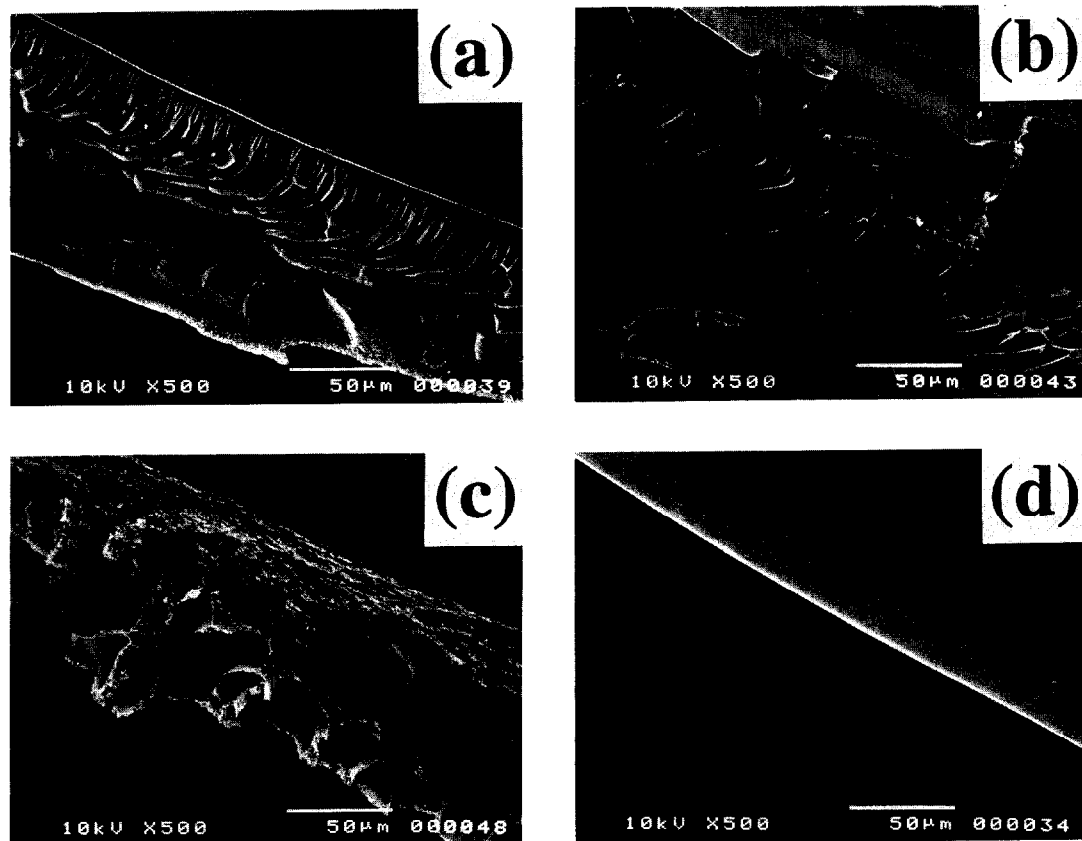


Fig. 6. SEMs of Radel A/PVP membranes. (a) 20 wt.% Radel A/10 wt.% PVP; (b) 20 wt.% Radel A/20 wt.% PVP; (c) 20 wt.% Radel A/25 wt.% PVP; and (d) 25 wt.% Radel A/25 wt.% PVP.

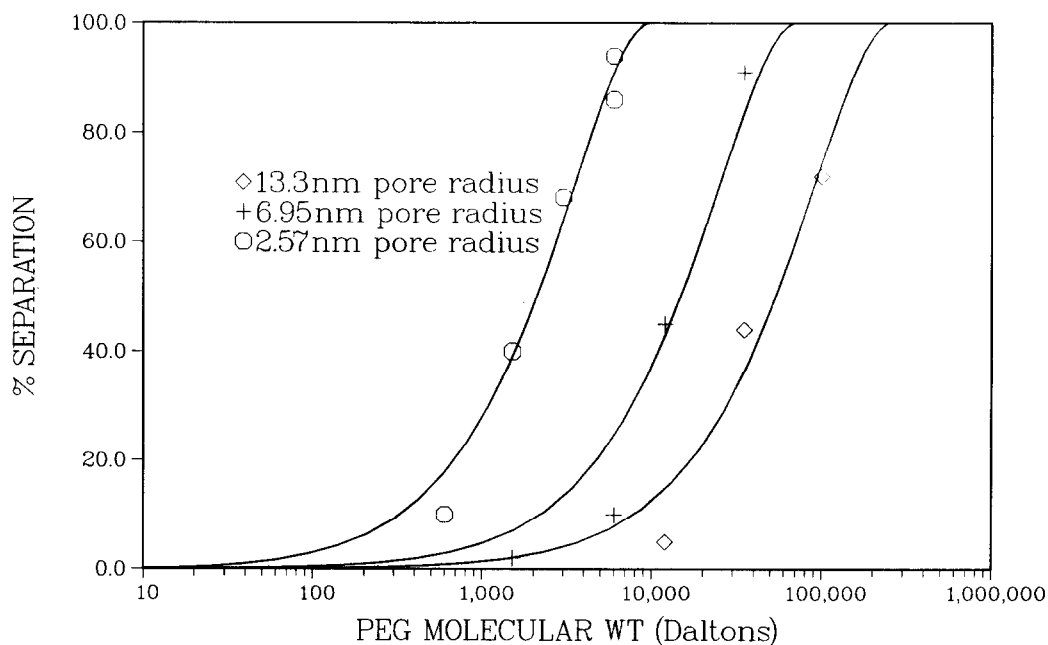


Fig. 7. Predicted and actual separation data for three Radel A/PVP membranes. (\diamond) 20 wt.% Radel A/0 wt.% PVP, (+) 20 wt.% Radel A/15 wt.% PVP, and (\circ) 25 wt.% Radel A/25 wt.% PVP.

roscopic structures is related to the PVP concentration and to the casting solution viscosity. The size of the finger-like voids increases with increasing PVP concentration [Fig. 6(a)–Fig. 6(c)]. However, at high Radel A and PVP concentrations, the finger-like structures are absent [Fig. 6(d)] and instead the sub-surface has a spongy structure. This sponge-like support structure can be attributed to the high viscosity of the casting solution. In general, the formation of such macroscopic structures is a function of the exchange rate between water and the solvent during the gelation step. A fast rate of exchange favors the formation of finger-like voids and a slow rate results in sponge layers. This rate is in part governed by the viscosity of the casting solution. The theory of membrane formation with respect to casting solution composition and viscosity can be found in the literature [12,13].

Some typical experimental separation data and predicted separation curves for three dif-

ferent membranes are shown in Fig. 7. The membranes were cast from a 20 wt.% Radel A solution, a 20 wt.% Radel A/15 wt.% PVP solution and a 25 wt.% Radel A/25 wt.% PVP solution. The calculated average pore sizes for those membranes were 13.3 nm, 6.95 nm and 2.57 nm, respectively. The fitted model adequately represents the experimental results for a wide range of pore sizes.

The ability of a membrane to provide a given separation can be described by its average pore size, which in turn can be related to the casting solution composition. This relationship is shown in Figs. 8(a) and 8(b) as a function of the Radel A and PVP concentration. Radel A/PVP/NMP casting solutions can produce membranes with an average pore radius of less than 3 nm. The contour plot shows that the average pore size is largely related to the concentration of Radel A alone for concentrations of PVP \leq 10 wt.%. For PVP concentrations greater than 10 wt.%, membrane pore sizes can be re-

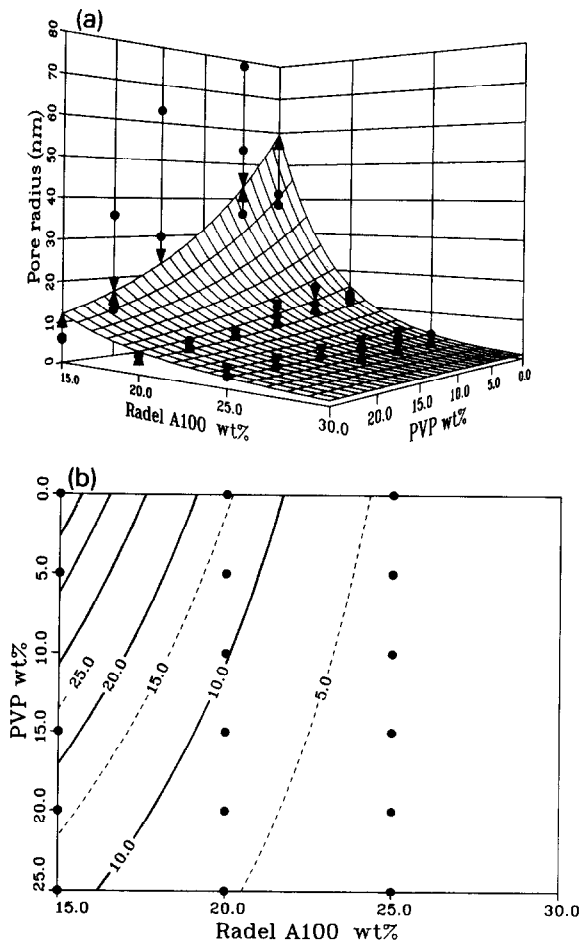


Fig. 8. (a) Membrane pore radius as a function of Radel A and PVP concentration. Surface obtained from a polynomial fitted to the experimental data (\bullet) using sum of squares of residuals fitting. (b) Contour plot representation of Fig. 8(a).

duced by increasing either Radel A or PVP concentrations.

Figures 9(a) and 9(b) show the effect of the casting solution composition on the pore density to pore length ratio. The contours show significant effects on $n/\Delta xA$ by adding PVP to the casting solution. High $n/\Delta xA$ (i.e. high productivity) for a given pore size could only be achieved with high concentrations of PVP. For example, a casting solution of 25 wt.% Radel A/5 wt.% PVP and one of 20 wt.% Radel A/20

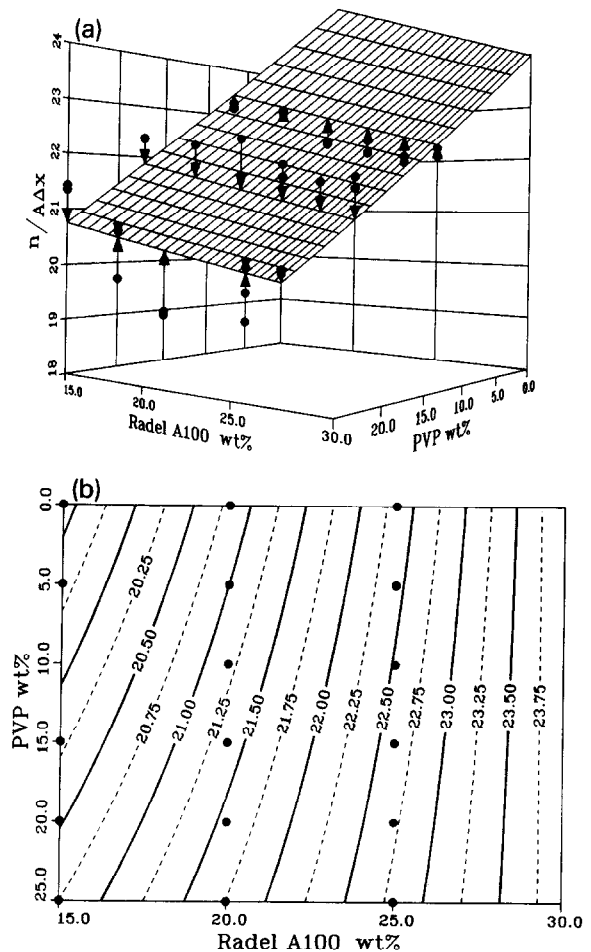


Fig. 9. (a) $n/\Delta xA$ as a function of composition. Surface obtained from a polynomial fitted to the experimental data (\bullet) using sum of squares of residuals fitting. (b) Contour plot representation of Fig. 9(a).

wt.% PVP produce membranes with pore sizes of 4.6 nm and 4.3 nm, respectively. The $n/\Delta xA$ for the two membranes are $0.8 \times 10^{22} \text{ m}^{-3}$ and $1.4 \times 10^{22} \text{ m}^{-3}$, respectively. Similar behavior has been found when PVP is present in other polysulfone membrane casting compositions [14-16].

A graphical method for comparing membrane performance is to plot $n/\Delta xA$ as a function of the pore radius. This allows one to determine which membranes would have the

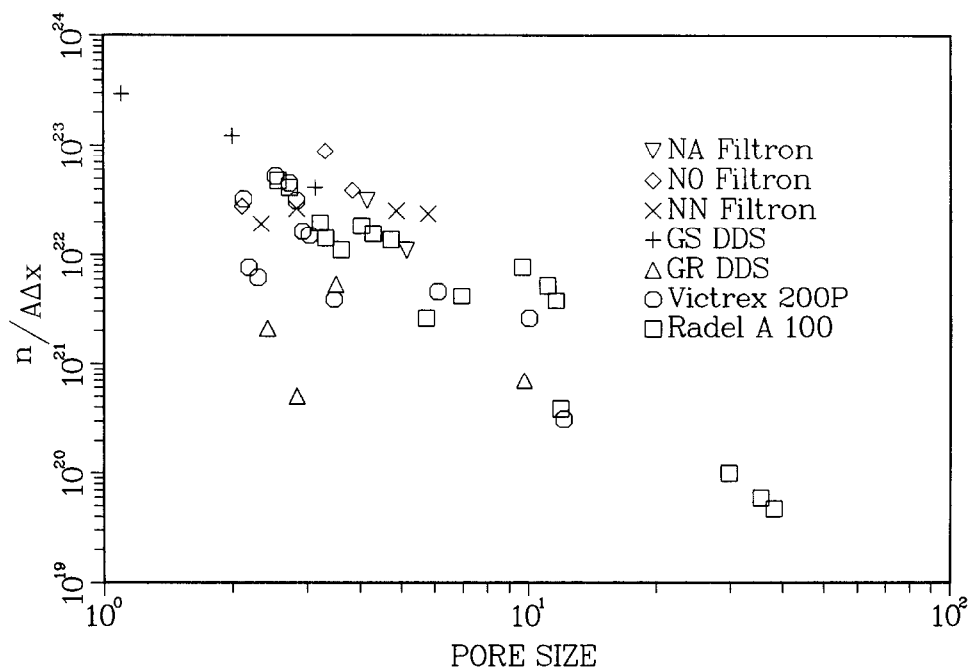


Fig. 10. A plot of $n/\Delta xA$ as a function of the pore radius for Radel A/PVP membranes, commercial polysulfone membranes and Victrex PES/PVP membranes.

higher permeation rate for a given pore size. Figure 10 compares the performance of commercial membranes, Radel A/PVP and Victrex PES/PVP membranes. The performance for commercial polysulfone membranes from Filtron and DDS have previously been reported [5]. We have also investigated the membrane characteristics of cast Victrex PES/PVP membranes [16]. In this work, Radel A and Victrex membranes were found to have similar performance characteristics. This is not unexpected considering the structural similarities as well as comparable physical and chemical properties between the two polymers.

Conclusions

NMR measurements showed that the chemical structure of Radel A100 is a copolymer consisting of repeating units of polyethersulfone and small amounts of polyetherethersulfone

(ratio 1:0.18). The small amount of PEES present in the copolymer does not significantly change the physical or membrane properties compared to commercial polyethersulfone. Membranes of pore sizes ranging from 2 nm to 40 nm were made from Radel A/PVP/NMP casting solutions. The macro-membrane morphology, average pore size and permeability were controlled by adjusting the membrane casting composition and casting solution viscosity. Radel A provides membrane manufacturers with additional flexibility in the choice of available membrane materials.

Acknowledgements

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List of symbols

A	area of the tested membrane coupon (m^2)
D_∞	diffusivity of solute in solution (m^2/sec)
f	separation
n	number of pores
Pe	Peclet number
Q	volumetric flow rate (m^3/hr)
r_p	pore radius (nm)

Greek

Δx	effective length of the membrane pore (m)
ξ	ratio of restricted diffusion within the pore to free diffusion in bulk solution
η	viscosity of water (Pa-sec)
χ	global steric parameter
ΔP	pressure drop across the pore (kPa)

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