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Separation Science and Technology
Publication details, including instructions for authors and subscription information:
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To cite this Article Wang, Jinwen , Yue, Zhongren and Economy, James(2009) 'Solvent Resistant Hydrolyzed Polyacrylonitrile Membranes', Separation Science and Technology, 44: 12, 2827 — 2839
To link to this Article: DOI: 10.1080/01496390903135923
URL: http://dx.doi.org/10.1080/01496390903135923

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Separation Science and Technology, 44: 2827–2839, 2009
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ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496390903135923

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Abstract: We have studied the solvent resistance of polyacrylonitrile membranes hydrolyzed by NaOH. The hydrolyzed polyacrylonitrile membranes provide a candidate material for either the selective layer or the supporting layer for the solvent resistant composite membrane. The origin of this solvent resistance is summarized. Polyacrylonitrile (PAN) cation-exchange NF membranes were prepared from ultrafiltration (UF) membranes through a convenient way developed recently by us. Several explanations for the difference of various solvent permeability coefficients of the cation-exchange PAN NF membrane were also suggested. Relatively high permeability was observed for the solvents with a benzene ring structure. Furthermore, products of their viscosities and permeability, μL_P^0, are close to a constant.

Keywords: Nanofiltration membrane, polyacrylonitrile, pure solvent permeability coefficients, solvent resistance

INTRODUCTION

Although polymeric membranes are primarily used with aqueous systems, numerous separation technologies for solutions polluted by organic

Received 1 December 2008; accepted 12 May 2009.
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solvents could be optimized by the application of solvent resistant polymeric membranes. The rapidly expanding interest in the separation of low molecular weight substances from nonaqueous solutions by membrane technologies also demands solvent-stable membranes. In addition, the substrate layer of thin film composite membranes, usually polysulfone, is attacked by many solvents, which limits the deposition of a permselective, ultra-thin membrane from solvent systems (such as DMF, NMP) and limits the development of new RO/NF membranes (1,2).

Polyacrylonitrile (PAN), because of its high degree of solvent resistance, is widely used as an UF membrane. Due to its highly hydrophilic properties, it has been known as a low fouling membrane for aqueous filtration. Compared to other polymer materials, PAN also has good resistance against chlorine (3). Several approaches have been reported to develop PAN membranes with improved chemical stability not only to common solvents but also to polar aprotic solvents like DMF, DMAc, and NMP. These approaches used systems such as PAN/metal ethoxides (4), PAN/sodium hydroxide with heating (5,6), poly(acrylonitrile-*co*-vinylacetate)/ hydroxylamine/sodium carbonate/ cyanuric chloride (7), and poly(acrylonitrile-*co*-glycidylmethacrylate) or poly(acrylonitrile-*co*-vinylbenzylchloride)/ multifunctional amines (8–10) to form crosslinked networks. Although Linder et al. (5,6) mentioned that the PAN became solvent resistant after the treatment with NaOH, they emphasized the use of heating and crosslinking agents. They did not propose the potential mechanism for solvent resistance resulting from the hydrolysis, either. Here we studied the solvent resistance of the PAN membranes hydrolyzed at room temperature by NaOH more thoroughly. By this simple method we can develop NF/UF membranes or substrates with excellent solvent resistance for TFC RO/NF membranes. We also summarize the origin of this solvent resistance (11–13).

In addition, cation-exchange PAN NF membranes were easily prepared from hydrolyzed PAN UF membranes according to a method we developed previously (14). Permeability of different solvents through the cation-exchange PAN NF was studied. Some explanations for the difference of various solvent permeability coefficients of the cation-exchange PAN NF membrane were also suggested.

EXPERIMENTAL

Reagents

PAN (homopolymer, $T_g = 85°C$, average M_w 150,000), polyvinylpyrrolidone (PVP, $M_w = 29{,}000$), and DMF were obtained from Aldrich. All the salts were used as received from Fisher Scientific. Methyl orange and

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bromothymrol blue were received from Alfa Aesar, disperse red from MP Biomedicals, LLC, and direct yellow 50 (dye content 40%) from Aldrich. All the solvents were used as received.

Preparation of Asymmetric PAN UF Membrane

The UF membranes were prepared by the phase separation technique using water as a coagulant. PAN was used as a membrane material and PVP as an additive to make the membrane more porous. PAN and PVP powder were dissolved at 80–90°C with stirring in DMF to form a 15:5 wt% PAN: PVP casting solution. The solution was cast onto a Hollytex® polyester nonwoven fabric using a laboratory membrane-casting machine (Separation Systems Technology, USA). The nascent membrane was immersed in a room temperature tap water coagulation bath without evaporation of solvents in the air. After precipitation, the membrane was kept in a water bath for several days and then washed with deionized water before carrying out further experiments.

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Preparation of Asymmetric Cation-Exchange PAN NF Membrane

The cation-exchange PAN NF membrane was prepared via our previous method (14). The PAN UF membrane was hydrolyzed with 1 M NaOH at room temperature for 24 hours. After saturation with 30 wt% $ZnCl_2$ solution, the membrane was heat treated in air at 110°C for 10 minutes. Following heating, the membrane was allowed to cool to room temperature in air. By soaking the membrane in a very dilute HCl aqueous solution (pH = 3–4), $ZnCl_2$ was removed and a semi-transparent PAN membrane that was 2–3 times thinner was formed. The color of the hydrolyzed yellowish red PAN membrane turned to yellowish white. Following that, the membrane was treated with a dilute NaOH solution (pH = 8–9) to convert it into a NF membrane with highly dense pore surface functional groups (–COONa, –CN and $–CONH_2$).

Measurement of Swelling Ratios of Hydrolyzed PAN UF Membranes in DMF

The degree of swelling of hydrolyzed PAN membranes was determined by the difference in lengths (L) and thickness (T) before and after immersion in DMF. PAN membranes prepared in section 2.2 were hydrolyzed in 1 M NaOH for various times at room temperature and washed with DI water until becoming neutral. The typical size of membranes before swelling

is around $50\,\text{mm} \times 5\,\text{mm} \times 0.5\,\text{mm}$ (Length × Width × Thickness). The hydrolyzed PAN membranes with and without the polyester non-woven fabric were then immersed in DMF for 72 hours at room temperature. The lengths and thickness of swollen membranes were quickly measured. The swelling ratio of the membrane was calculated from the following equation:

$$\text{Length swelling ratio} = (L_S - L_O)/L_O \times 100\%$$

$$\text{Thickness swelling ratio} = (T_S - T_O)/T_O \times 100\%$$

L_S and L_O are the lengths of membranes, T_S and T_O are the thickness of membranes after and before swelling, respectively.

Membrane Performance Measurement

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The membranes were cut into disks for use in a filtration cell (Sterlitech™ HP4750 Stirred Cell). A standard magnetic stirrer (Corning Stirrer/Hot Plate, Model PC-420) was used and the stirrer speed was set to achieve a reasonable rate of stirring. Rejection was determined using a NaCl solution. The salt solution flux and salt rejection were measured at 200 psig and room temperature. The feed concentration was typically 2000 mg/L in pure DI water. The permeated samples were collected after a few minutes and the concentration of permeates were determined, using a Corning pH/ion analyzer 455.

Pure solvent permeability coefficients of cation-exchange NF membranes were measured by applying pressures of 6.89, 10.34, and 13.79 bars.

The dye molecules used to characterize the membrane performance in methanol was selected on the basis of easy detection, charge, and size. The concentration of disperse red (DR), methyl orange (MO), bromothymol blue (BTB), and direct yellow 50 (DY) were recorded on a Varian Cary 5G spectrophotometer. The molecular weight, the charge of the dyes, and the wavelength used for their detection were listed in Table 1. The feed concentration of these dyes in methanol was 1.0 g/L. The cation-exchange PAN NF membrane in this test had a 30% rejection of 2 g/L NaCl with a flux of $0.3 \times 10^{-3}\,\text{m}^3\,\text{m}^{-2}\,\text{day}^{-1}$at a pressure of 13.79 bars. The experiment in methanol was performed at a pressure of 13.79 bars with a flux of $1.2 \times 10^{-3}\,\text{m}^3\,\text{m}^{-2}\,\text{day}^{-1}$.

Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR/FTIR)

ATR/FTIR spectra were collected in the range 4000–600 cm^{-1}, on a Nexus 670 FT-IR (Thermo Electron Corporation, Madison, WI) with a Golden

Table 1. Rejection of various dyes of cation-exchange PAN NF membranes

Dyes	Disperse red	Methyl orange	Bromothymol blue	Direct yellow 50
MW (g/mol)	314	327.3	624.39	956.8
Charge	0	−1	0	−4
Max wavelength (nm)	570	421	420	398
Rejection (%)	24	20	43	75

Gate™ MKII Single Reflectance ATR (Specac Inc., Woodstock, GA). The spectrometer was installed with a deuterated triglycine sulfate-potassium bromide (DTGS-KBr) detector and KBr beamsplitter. Spectra collection was performed using FT-IR software (OMNIC, Thermo Electron Corporation, Madison, WI) and analyzed using spectrum software (KnowItAll Informatics System 5.0 Academic Edition, Bio-Rad Laboratories, Inc). Spectra were recorded by positioning the samples on a cell platform operating at room temperature (64 scans, 4 cm^{-1} resolution).

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RESULTS AND DISCUSSION

Solvent Resistance of Hydrolyzed PAN Membranes

In this study, we found that the PAN membrane became resistant to all common organic solvents at room temperature after hydrolysis with NaOH. The solvent resistance of the cation-exchange PAN NF membrane is demonstrated in Fig. 1. The membrane was soaked in DMF for a week. Both flux and rejection decreased a small amount at first; but then the membrane performance became stable. In addition, heating the membrane in DMF at 80°C for 8 hours showed some change in performance, but no dissolution was observed.

The effect of hydrolysis of PAN UF membranes was also studied. Figure 2 shows the solvent resistance of PAN UF membranes hydrolyzed by NaOH for different times. The decrease in the pure water permeability at the beginning might be due to the swelling of the membrane in DMF. The longer the time of hydrolysis, the smaller this decrease is. On the other hand, after the decrease, the permeability increases a small amount for the membrane hydrolyzed for 8 hours and 12 hours. This increase becomes less obvious for the membrane hydrolyzed for a longer time. For example, the pure water permeability of the membrane hydrolyzed for 24 hours even becomes stable after soaking in DMF for a short time. From the above observations, the hydrolysis is probably accompanied by some kind of crosslinking reactions. The reason for the increase of the

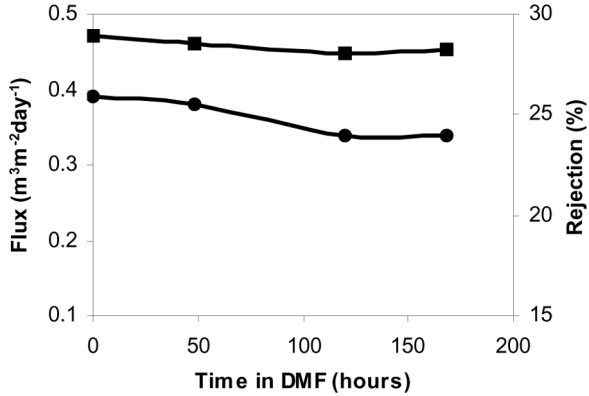

Figure 1. Effects of soaking time in DMF on flux (●) and rejection (■) of PAN cation-exchange NF membranes.

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permeability for membranes hydrolyzed for 8 and 12 hours might be due to the dissolution of some uncrosslinked polymer chains. As the hydrolysis progresses, more crosslinks are formed and the membrane becomes more resistant to DMF, which results in the less obvious increase of the pure water permeability.

The degree of swelling of hydrolyzed PAN membranes in DMF is shown in Fig. 3. Figure 3(a) is the degree of swelling of membranes with

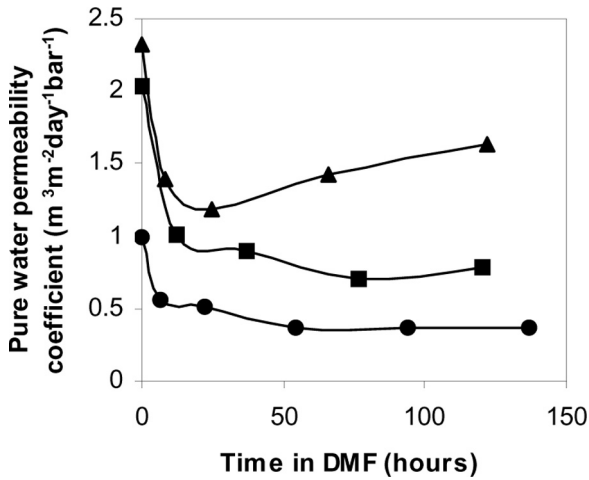

Figure 2. Effects of hydrolysis on solvent resistance of PAN UF membranes in DMF. ▲ – 1M NaOH for 8 hours, ■ – 1M NaOH for 12 hours, ● – 1M NaOH for 24 hours.

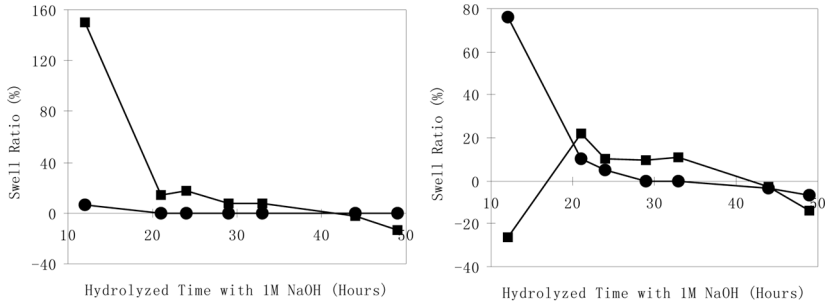

Figure 3. Effects of hydrolysis time on the length swell (●) and thickness swell (■) of PAN UF membranes in DMF for 72 hours with (a) and without (b) non-woven polyester support.

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the polyester support. In general, the swelling ratio in length decreases with hydrolysis. As expected, the degree of swelling in length is very small, even zero, due to the restraints from the polyester support. In the direction of thickness, the membrane swells and the swell ratio is almost stable between 21 and 33 hours hydrolysis. Interestingly, after about 40 hours of hydrolysis, there are negative swelling ratios, i.e., the membranes shrink. In Fig. 3(b) is shown the degree of swelling of membranes without the polyester support. Again, the swelling ratio in length decreases with the hydrolysis. In the direction of thickness, the swell ratio is close to a constant for membranes hydrolyzed between 24 and 33 hours. After 40 hours of hydrolysis, membranes shrink in both directions. This phenomenum indicates that the crosslinking degree increases as the hydrolysis progresses.

Mechanism of Solvent Resistance of Hydrolyzed PAN Membranes

Based on the literatures (11–13), there could be two possible crosslinking reactions (See Fig. 4). Romanova et al. (11) monitored the alkaline hydrolysis of a dilute PAN solution through UV spectra. They observed that a new adsorption peak appeared between 300 nm and 395 nm with continued hydrolysis, which indicated the formation of conjugated sequences. At the initial stage of hydrolysis, PAN will form $(–C{=}N–)_n$ conjugated sequences as intermediates. The yellowish red color of PAN UF membranes suggests the presence of conjugation. When the hydrolysis continues, the conjugated sequences will be further hydrolyzed into sodium acrylates and amides. Since the PAN membrane is only partly hydrolyzed, some $(–C{=}N–)_n$ conjugated sequences will be definitely left

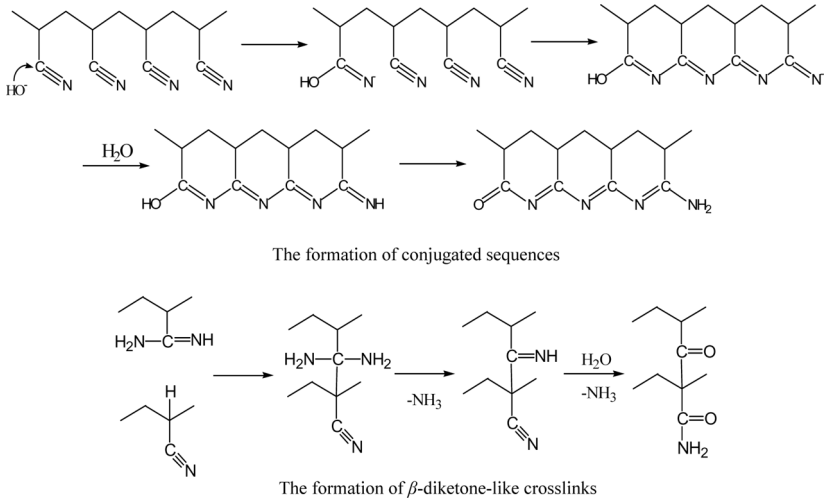

Figure 4. Two possible crosslinking reactions during hydrolysis.

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within the bulk of the membrane, which contributes to the solvent resistance. The other possible crosslinking reaction might be the formation of β-diketone-like crosslinks proposed by Kudryavtsev et al. (12) where an amidine intermediate interacts with a mobile α-H atom of acrylonitrile group on another PAN chain. The existence of amidine was confirmed by ^{13}C NMR spectroscopy (13). On the other hand, by kinetic measurements, Kudryavtsev et al. estimated the ratio of the initial rate constants for the polyacrylonitrile and polyacrylamide alkaline hydrolysis, which was found to be of the order of 10^{-1}. This indirectly proved the formation of β-diketone-like crosslinks. Alternatively, the potential to form ionic like structures leads to a kind of crosslinking, i.e., carboxylate arising from Na^+ ionic crosslinks with carboxylate groups.

Rejection of Different Dyes in Methanol

The permeation data for different dyes are listed in Table 1. Evidently, molecular size has the principal effect on the rejection. The cation-exchange PAN NF membrane had a higher rejection for dyes with larger molecular size. The rejection of DR and MO was similar to each other, which showed that the charge of dyes might have little effect on the permeation. The reason for this phenomenon might be that the cation-exchange PAN NF membrane became neutral in methanol in comparison with the membrane in water where the –COONa is easily dissociated.

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Permeability of Pure Solvents Through the Cation-Exchange PAN NF Membranes

Permeability of some pure solvents at room temperature through the cation-exchange PAN NF membranes is listed in Table 2. For the nonsolvents of PAN/partially hydrolyzed PAN including hexane, acetone, THF, chloroform, and different alcohols, the permeability generally decreases with increasing of the viscosity of the bulk solvent. The viscosity within nanopores of these solvents is unavailable in the literature. Wessling et al. (17) and Vankelecom et al. (18) also pointed out the similar effect of the viscosity on the permeability. Besides the viscosity, the molecular size and shape might also play a role, i.e., the smaller the molecular dimensions, the larger the permeability. For good solvents including DMF and DMAc, the pores within the membrane might become smaller due to the swelling of PAN/partially hydrolyzed PAN, which makes the permeability much lower than with nonsolvents. It is interesting that very high permeability was observed with solvents containing aromatic ring structures such as benzene and its derivatives. Water is a solvent between good solvents and nonsolvents and hence the permeability is between those of good solvents and nonsolvents. Another trend in the table is that the products of permeability and viscosity of nonsolvents are all in the range of 2.21 (1,4-Dioxane) and 3.26 (Toluene) except cyclohexane whose dimension might be too large. This trend is obvious for benzene, toluene, and xylene, i.e., the product is close to 3.2. The explanation for this phenomenon can be found from the Hagen–Poiseuille equation. As we know, the simplest model to describe membrane performance considers a membrane as a number of parallel cylindrical pores, the length of each pore being equal or almost equal to the thickness of the top layer of the membrane. This model describes the flux through micropores in an ideal situation, i.e., with uniformly distributed and evenly sized pores in the membrane, with no fouling, negligible concentration polarization, hydrophobicity, surface tension, etc. The volume flux through these pores may be described by the Hagen–Poiseuille equation for viscous flow (19):

$$J = \mathrm{L}_\mathrm{p}^0 \Delta \mathrm{P}$$

$$\text{Pure Water Permeability Coefficient: } \mathrm{L}_\mathrm{p}^0 = \varepsilon r^2/(8\mathrm{L}\mu\tau)$$

Where J is the solvent flux through the membrane, ε the surface porosity of the membrane, r the average pore radius, ΔP the applied transmembrane pressure, μ the viscosity of the liquid permeating the membrane, L the membrane thickness, and τ is the tortuosity factor. Therefore, $\mu \mathrm{L}_\mathrm{P}^0$ should be a constant only determined by membrane

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Table 2. Pure solvent permeability of cation-exchange PAN NF membranes

Solvents	Water	Chloroform	Methanol	Ethanol	2-Propanol	Acetone	DMF	DMAc
L_p^0 ($10^{-3}\,m^3m^{-2}h^{-1}bar^{-1}$)	1.0	5.3	4.2	2.4	1.5	8.8	0.09	0.07
Viscosity μ (cP,25°C)	0.890	0.540	0.551	1.07	2.07	0.306	0.794	0.927
μL_p^0	0.89	2.86	2.31	2.57	3.10	2.69	0.072	0.065

Solvents	Hexane	Cyclohexane	Benzene	Toluene	*o,m,p*-Xylene	1,4-Dioxane	THF
L_p^0 ($10^{-3}\,m^3m^{-2}h^{-1}bar^{-1}$)	10	1.4	5.3	5.9	5.0	1.8	5.9
Viscosity μ (cP,25°C)	0.294	0.912	0.603	0.553	0.63	1.255	0.460
μL_p^0	2.94	1.28	3.20	3.26	3.15	2.21	2.70

Note: Viscosity from ref. (15,16).

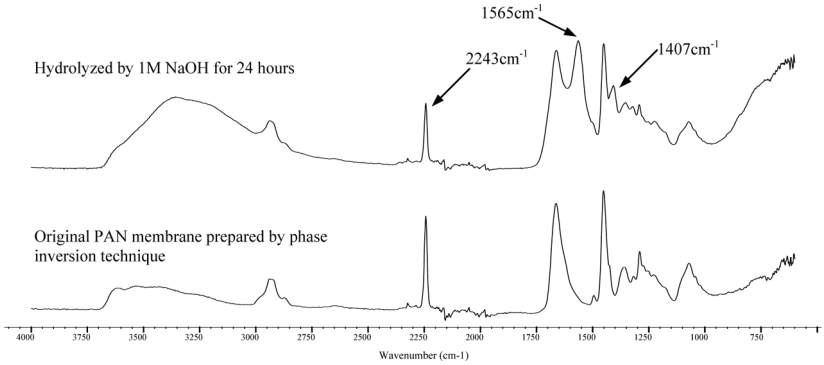

Figure 5. ATR/FTIR of PAN membranes.

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properties. In the review written by Bhanushali et al., a similar trend was also observed (20).

The ATR-FTIR spectra of membranes before and after hydrolysis are shown in Fig. 5. The new peaks at 1565 cm^{-1} and 1407 cm^{-1} correspond to the –C=O asymmetric and symmetric stretching vibrations in the –COONa groups. Since the peak at 2243 cm^{-1} for –C≡N does not change significantly, only a fraction of the –C≡N groups were hydrolyzed.

CONCLUSION

The PAN membranes became resistant to all common organic solvents including DMF, DMSO, and NMP after hydrolysis, which provides a candidate material for either the selective layer or the supporting layer for a solvent resistant composite membrane. Several crosslinking mechanisms were summarized. Some explanations for the difference of various solvent permeability coefficients of the cation-exchange PAN NF membrane were suggested. Very high permeability through the cation-exchange PAN NF membrane was observed with solvents containing aromatic ring structures such as benzene and its derivatives. The product of permeability and viscosity of benzene and its derivatives is almost a constant, i.e., 3.2.

ACKNOWLEDGEMENTS

This work was supported by The WaterCAMPWS, a Science and Technology Center of Advanced Materials for the Purification of Water with Systems under the National Science Foundation agreement number

CTS-0120978. The ATR/FTIR were measured with the help of David Ladner in Professor Timm Strathmann's group at the University of Illinois at Urbana-Champaign.

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