Synthesis and Characterization of Polymer-Filled Nonwoven Membranes

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ABSTRACT: Polymer-filled nonwoven membranes were prepared by filling the open pores of nylon nonwovens with poly(2-acrylamido-2-methyl-1-propanesulfonic acid) (PAMPS). PAMPS was synthesized via radical polymerization and crosslinked to prevent its dissolution in water. PAMPS-filled nylon nonwoven membranes showed enhanced dimensional stability and mechanical properties when compared with PAMPS membranes without nonwovens. The conductivities of PAMPS-filled nylon nonwovens were slightly lower than those of PAMPS membranes. Compared with PAMPS membranes without nonwoven hosts, both linear and crosslinked PAMPS-filled nylon nonwoven membranes exhibited lower vapor permeabilities for water, methanol, acetone, and dimethyl methylphophonate (DMMP). In addition, crosslinked PAMPS-filled nonwoven membranes presented high permselectivity on DMMP over water, which is critical for chemical protection application. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 2568–2575, 2011

Key words: barrier; composites; fibers

INTRODUCTION

Functional polymer membranes that are capable of separating liquid and/or vapor mixtures have a variety of potential applications including chemical protection, water treatment, rare element recover, catalyst, drug delivery, bioseparation, pharmaceutical purification, environmental processes, materials synthesis, ultrafiltration, batteries, and fuel cells.[1–7] However, traditional single-component functional polymer membranes often have poor mechanical strength and stability, which limit their use in many practical applications, and hence, reinforced polymer composite membranes have been investigated. So far, several different types of reinforcements have been incorporated into polymer membranes to improve their mechanical properties, and thermal and dimensional stabilities. For example, porous inorganic[8–11] and nonconducting polymer[12–16] substrates have been used as the hosting materials to reinforce functional polymers. Compared with single-component polymer membranes, these polymer-filled porous membranes have the advantages of being able to tune both the functional and mechanical properties by controlling the volume fraction of the polymer occluded in their pores. However, the porous host experiences large stress while suppressing the swelling of functional polymers. As a result, the porous hosts often lose structural integrity after prolonged usage, especially in applications where membranes face repeated hydration and dehydration.

Nonwoven fabrics are made directly from web of fibers through interlocking or bonding, and they are promising candidate for reinforcing functional polymers because of their excellent mechanical properties. When nonwoven fabrics are used as the hosting material to reinforce functional polymers, they can provide good mechanical strength and structural integrity without sacrificing the functionality of occluded polymers. At the same time, nonwoven fabrics, consisting of entangled polymer fibers, are flexible and can withstand repeated swelling and contraction of functional polymers. In addition, the large void volume inside nonwovens provides a high capacity to host a large amount of functional polymers.

Poly(2-acrylamido-2-methyl-1-propanesulfonic acid) (PAMPS) is a functional polymer that contains sulfonic acid terminated side chains and shows strong ion-binding properties and high conductivity, resulting from the high dissociation constant due to sulfonic acid groups.[17] PAMPS membranes have been used as ion exchange membranes,[18–20] humidity sensors,[21] nanofiltration films,[22] and polymer electrolytes in fuel cells.[23] However, PAMPS is mechanically weak and undesirably swelled in aqueous solutions. In this work, nylon nonwoven fabrics were used as the host material for PAMPS to improve mechanical

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properties of PAMPS membranes. PAMPS-filled nylon nonwoven membranes were characterized to explore their applications.

EXPERIMENTAL

Materials

Monomer 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS), initiator 2,2′-azobis(2-methylpropionitrile) (AIBN), crosslinking agent ethylene glycol diacrylate (EGD), and solvents including dimethylsulfoxide (DMSO), methanol, acetone, and dimethyl methylphophonate (DMMP) were purchased from Sigma–Aldrich. Nylon nonwoven fabrics were provided by Nonwoven Cooperative Research Center at North Carolina State University. These nonwoven fabrics were first fabricated as the island (nylon) in sea (polylactic acid) structure using a spunbonding technique, but polylactic acid was removed before usage to form the nylon nonwoven structure. The thickness of nylon nonwoven fabrics was 0.2 mm, and the basis weight and solidity of nylon nonwoven fabrics were 0.06 kg/m^2 and 40%, respectively.

Preparation of linear and crosslinked pamps-filled nonwoven membranes

PAMPS was synthesized by free radical polymerization using AIBN as an initiator. AMPS and AIBN were dissolved in DMSO and heated at 60°C. After 4 h, the mixtures were precipitated in acetone and dried in vacuum oven to collect PAMPS. The collected polymer was then dissolved in water and cast as free-standing PAMPS membranes. PAMPS-filled nonwovens were prepared by soaking nylon nonwovens in PAMPS aqueous solution and drying in vacuum oven.

Crosslinked PAMPS membranes were also prepared to overcome the undesirable swelling and dissolution of PAMPS in water. The crosslinking agent, EGD, was added in the mixtures of AMPS, AIBN, and DMSO to obtain crosslinked PAMPS. To prepare crosslinked PAMPS-filled nonwovens, weighed nylon nonwoven fabrics were soaked in the mixtures of AMPS, AIBN, EGD, and DMSO, which was placed between two Teflon plates, followed by heating at 60°C for 4 h and drying in vacuum oven.

The morphologies of nylon nonwoven fabric, PAMPS, and PAMPS-filled nonwoven membranes were examined using Hitachi S-3200 Scanning Electron Microscope.

Swelling ratio

The swelling behavior of PAMPS-filled nonwoven membranes was evaluated by measuring their swelling ratios (or solvent uptakes) in different solvents such as distilled water, methanol, and acetone. All measurements were carried out by soaking membranes in solvents at 25°C for at least 24 h. The swelling ratios (SR) were calculated by $\mathrm{SR}(\%) = \frac{\mathrm{W_2 - W_1}}{\mathrm{W_1}} \times 100$, where W_1 and W_2 were the membrane weights before and after soaking, respectively.

Mechanical properties

Tensile properties of nylon nonwovens and PAMPS-filled nonwoven membranes were measured using an Instron Universal Testing Machine at room temperature and 50% relative humidity (RH). The size of tensile specimens was 6 mm × 30 mm and the extension rate was maintained at 1 mm/sec.[24] Tensile strength, elongation, tensile modulus, and energy to break were calculated from the load-elongation curves.

Ionic conductivity

The conductivities of PAMPS and PAMPS-filled nonwoven membranes were measured by placing them in two-electrode conductivity cells, which were bathed in the water vapor with controlled temperature (30–70°C) and relative humidity (RH, 60–80%) in an environment chamber (Despatch Temperature-Humidity Test Chamber RTH-200-S). Electrochemical impedance spectroscopy (EIS, Gamry Reference 600 Potentiostat/galvanostat/ZRA) was used to determine the membrane resistances. The frequency range used was 100 – 100,000 Hz. The conductivities of membranes were calculated from the resistances using $\sigma = \frac{1}{R}\frac{L}{S}$, where R is the membrane resistance, L the membrane length, and S the membrane cross-sectional area.

Vapor permeability and permselectivity

The vapor permeabilities of PAMPS and PAMPS-filled nonwoven membranes were measured using the modified method based on the American Society for Testing and Materials (ASTM) E96–95 procedure. Membranes were placed inside the open-top caps of vials that were filled with solvents such as water, methanol, acetone, and dimethylmethylphosphonate (DMMP) (Fig. 1). The vials were placed in an environment chamber (35°C and 10 RH %), and their weight losses were recorded every 24 h. The vapor transfer rates of membranes were first calculated by $\mathrm{VTR} = \frac{\mathrm{G}}{\mathrm{t \times A}}$, where G is the weight loss (i.e., the weight of penetrant) at the steady state, t the time, A the membrane area exposed to vapor. Vapor permeabilities were then obtained using $\mathrm{VP} = \frac{\mathrm{VTR \times L}}{\Delta \mathrm{P}}$, where Δp is the vapor pressure differential across the membrane and L, membrane thickness. The permselectivities of membranes on DMMP over water, i.e., the ratios of DMMP permeability over water permeability, were calculated by $S = \frac{\mathrm{VP_{DMMP}}}{\mathrm{VP_{Water}}}$.

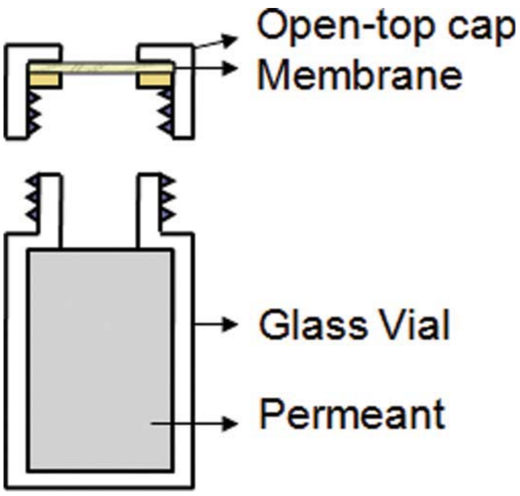

Figure 1 Schematic of vapor permeation testing cell. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

Membrane preparation

Nylon nonwoven fabric is durable and has interconnected open pores. Hence, polymer-filled nonwoven membranes can be prepared by filling the open pores of nylon nonwoven fabric with a functional polymer, PAMPS. Figure 2(a–c) shows SEM images of nylon nonwoven, linear PAMPS membrane, and linear PAMPS-filled nonwoven membrane. It is shown that, in linear PAMPS-filled nonwoven membrane, fibers of nylon nonwoven are completely covered by PAMPS, which occludes open pores of the nonwoven. After filling the nonwoven pores, the membrane weight increases by about 300% and the thickness increases by 200% to around 0.4 mm. The PAMPS content in the resultant composite membranes is around 75 wt %.

To overcome the high dissolution of linear PAMPS in water, PAMPS was crosslinked using a crosslinking agent, EGD. When the crosslinking degree of PAMPS is low (<20%), crosslinked PAMPS-filled nonwoven membranes still cannot maintain good integrity in water. However, when the crosslinking degree is greater than 35%, the membranes become brittle. Therefore, a crosslinking degree of 30% was selected in this work so that the resultant crosslinked PAMPS-filed nonwoven membranes can have balanced mechanical properties and dimensional stability. Figure 2(d,e) shows SEM images of crosslinked PAMPS and crosslinked PAMPS-filled nonwoven membranes (crosslink degree = 30%), respectively. It is seen that fibers in nylon nonwoven are covered by crosslinked PAMPS.

Swelling ratio

Most functional polymers, including crosslinked PAMPS, swell in solvents, which causes poor dimension stability and limited durability for membranes made from these functional polymers. Therefore, the

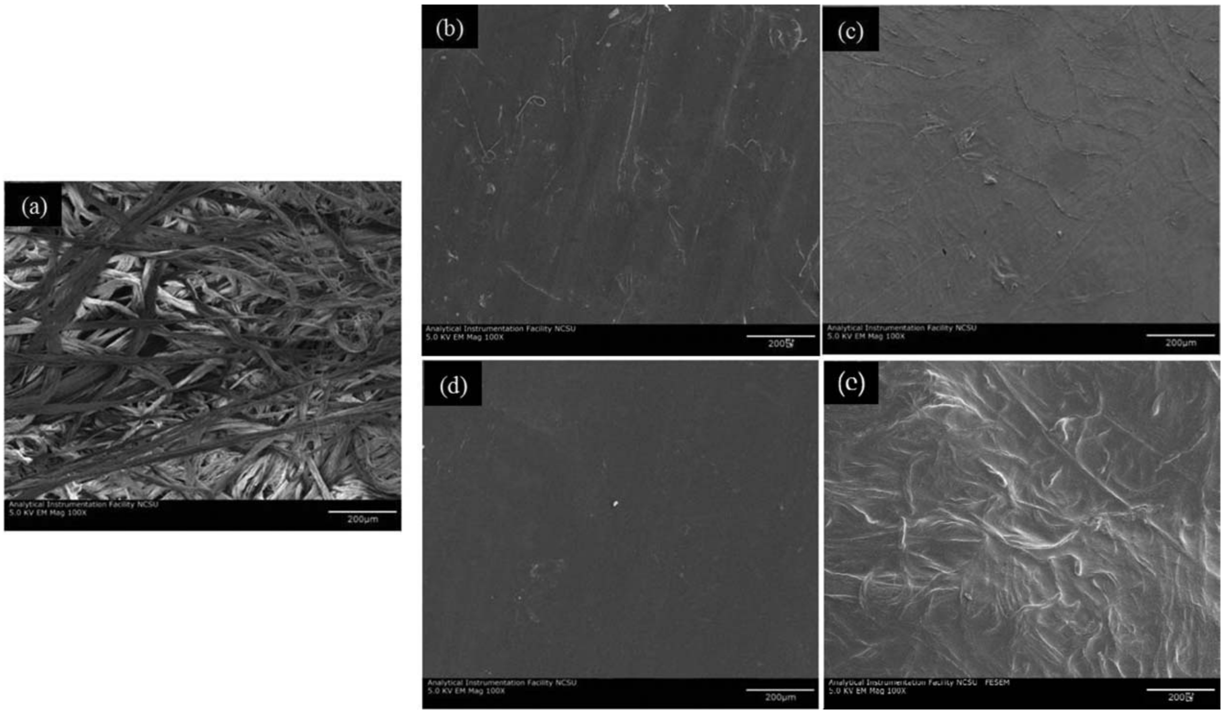

Figure 2 SEM images of (a) nylon nonwoven, (b) linear PAMPS, (c) linear PAMPS-filled nonwoven, (d) crosslinked PAMPS, and (e) crosslinked PAMPS-filled nonwoven membranes.

TABLE I
Swelling Ratios of Crosslinked PAMPS and Crosslinked PAMPS-Filled Nonwoven Membranes

	Crosslinked PAMPS	Crosslinked PAMPS-filled nonwoven
Water	119.0 ± 3.9	108.7 ± 22.7
Methanol	48.6 ± 5.1	26.3 ± 1.4
Acetone	20.5 ± 2.5	19.0 ± 1.4

severe swelling of functional polymers in solvents is one of the major problems faced by current polymer membrane technologies. Using porous substances to host functional polymers is an effective way to prevent the extreme swelling of polymer membranes.[13,15,16]

The swelling ratios of crosslinked PAMPS and crosslinked PAMPS-filled nonwoven membranes were measured in water, methanol, and acetone, and the results are shown in Table I. Linear PAMPS is dissoluble in water, and the swelling behavior of linear PAMPS could not be measured. From Table I, it is seen that both crosslinked PAMPS and crosslinked PAMPS-filled nonwoven membrane exhibit significantly higher swelling ratios in water than in other solvents, indicating that crosslinked PAMPS prefers to absorb water. The interaction between sulfonate and water via hydrogen bonds may promote the water absorption capability of PAMPS. From Table I, it is also seen that using nylon nonwoven to host PAMPS can reduces the membrane swelling ratio, especially in methanol, which is important for fuel cell application.

Mechanical properties

Nylon nonwovens are generally known to have good tensile strength, excellent heat shrinkage resistance, good air permeability, and high wear resistance, and hence, they are a good candidate for the reinforcement of PAMPS membranes. In PAMPS-filled nonwoven membranes, the interconnected open pores of nylon nonwovens are filled with functional PAMPS and the entangled nylon fibers provide mechanical strength and membrane integrity. Table II shows tensile properties of nylon nonwoven, linear PAMPS, linear PAMPS-filled nonwoven, and crosslinked PAMPS-filled nonwoven membranes. Nylon nonwovens show high flexibility while linear PAMPS membranes show high mechanical strength. It is seen that linear PAMPS-filled nonwoven membranes have higher tensile strength, larger Young's modulus, and greater energy-to-break than both nylon nonwoven and linear PAMPS membranes. In addition, the elongation at yield point increases after introducing nylon nonwovens into linear PAMPS membranes. Table II does not show the tensile properties of crosslinked PAMPS membranes because these membranes could not endure the pressure from the grips during tensile tests. PAMPS has a large amount of sulfonic acid groups, which lead to large interactions between polymer chains. After crosslinking, the movement of polymer chains is restrained, and this makes crosslinked PAMPS membranes brittle. However, using nylon nonwoven to host crosslinked PAMPS can significantly improve the membrane mechanical properties, although the resultant crosslinked PAMPS-filled nonwoven membranes still have lower tensile properties than other three membranes, as shown in Table II.

Proton conductivity

PAMPS shows excellent conductivity due to its sulfonic acid group, and this is an important characteristic in many applications, such as fuel cell electrolytes and sensors. When PAMPS is hydrated, protons can be dissociated from sulfonic acid groups and be transported across the membrane. Therefore, the main factors for determining the membrane conductivity are the amount and mobility of dissociated protons.[25]

Figure 3 shows typical EIS curves of PAMPS membranes at 70°C and 80 RH %. The membrane resistance can be obtained from the intersection of the spectrum curve with the real impedance axis. It is seen that introducing nylon nonwoven into linear PAMPS and crosslinked PAMPS increases their resistances. The ionic conductivities of these membranes at different temperatures and relative humidities were calculated based on the membrane

TABLE II
Tensile Properties of Nylon Nonwoven, Linear PAMPS, Linear PAMPS-Filled Nonwoven, Crosslinked PAMPS-Filled Nonwoven Membranes

	Nylon nonwoven	Linear PAMPS	Linear PAMPS-filled nonwoven	Crosslinked PAMPS-filled nonwoven
Strength (MPa)	0.16	0.20	0.26	0.08
Elongation at yield point (%)	53	15.0	44	8.6
Young's modulus (MPa)	110	150	450	82
Energy to break (kg-mm)	31	32	95	24

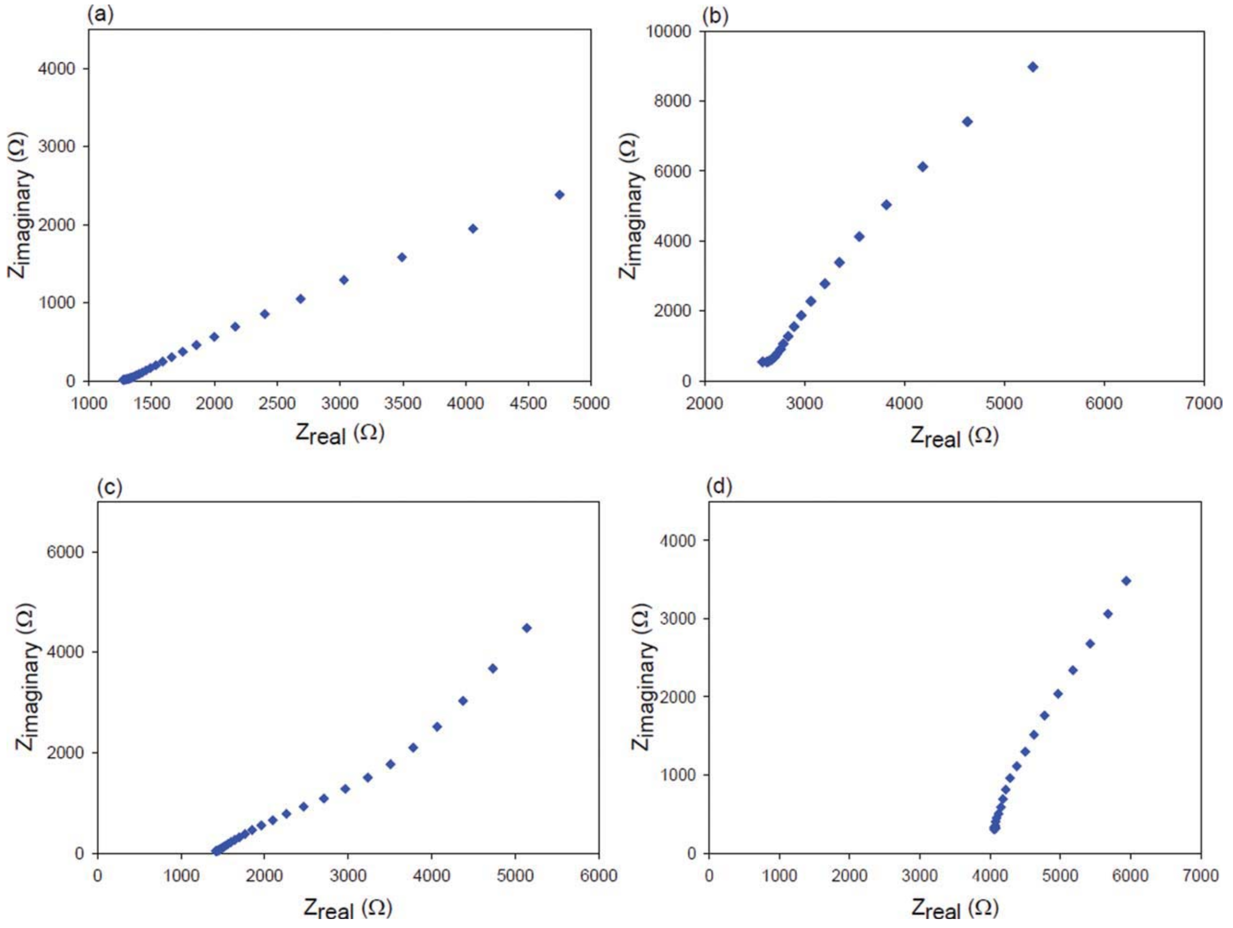

Figure 3 Typical impedance spectra of (a) linear PAMPS, (b) linear PAMPS-filled nonwoven, (c) crosslinked PAMPS, and (d) crosslinked PAMPS-filled nonwoven membranes at 70°C and 80 RH%. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

resistances and are shown in Figure 4. It is seen that linear PAMPS-filled nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes have higher conductivities at 80 RH% than at 60 RH% because more protons are dissociated from sulfonic acid groups at higher RH. However, linear PAMPS membranes without nonwoven host show similar conductivities at both relative humidities, indicating that a RH of 60% is high enough to fully hydrate linear PAMPS if the polymer chains are not constrained by nylon nonwoven. From Figure 4, it is also seen that linear PAMPS membranes exhibit higher conductivities than crosslinked PAMPS membranes. Qiao et al. also reported that the crosslinking of PAMPS using poly(vinyl alcohol) reduces the membrane conductivity.[26] The swelling of membrane materials is one important factor in determining their conductivities. Compared with linear PAMPS, crosslinked PAMPS membranes do not swell as much, resulting in lower ionic conductivities. Therefore, the crosslinking degree of PAMPS can be used to control the balance between the conductivity and dimensional stability of membranes.

The membrane conductivities are also affected by the temperature. As shown in Figure 4, the conductivities of all four membranes increase as temperature increases, indicating a positive temperature-conductivity dependence due to the enhanced mobility of protons at higher temperatures. However, compared to the conductivities of linear PAMPS membranes, those of crosslinked PAMPS membranes are shown to be less affected by the temperature change. This indicates that the enhancement of proton mobility at high temperatures is constrained by the formation of crosslinked network.

It must be noted that, compared with crosslinked PAMPS membranes without nonwoven host, crosslinked PAMPS-filled nylon nonwovens have lower conductivities over the entire temperature and RH ranges. However, the conductivity losses are not significant after considering the volume effect of nonconducting component, i.e., nonwoven host.

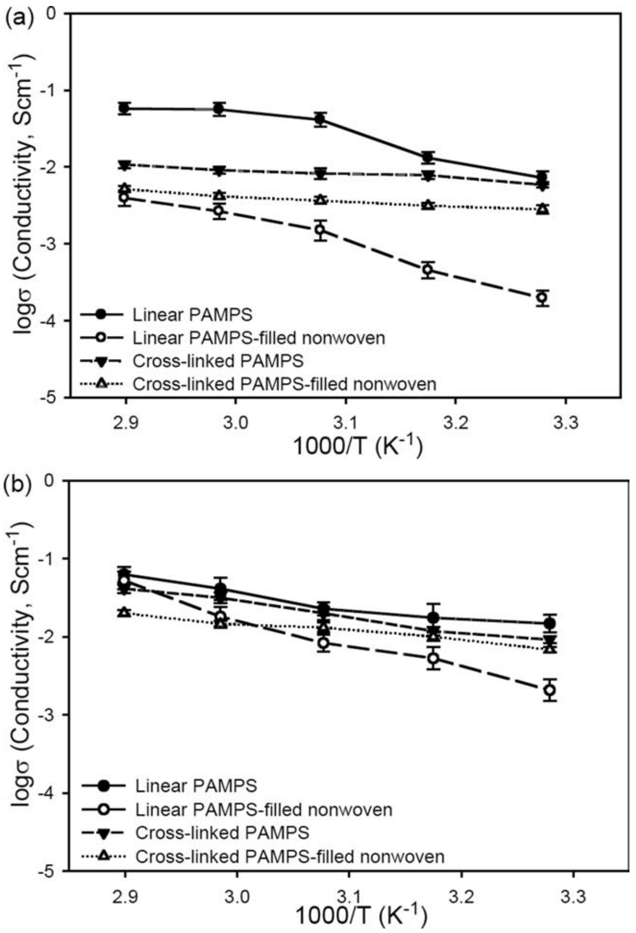

Figure 4 Proton conductivities of linear PAMPS, linear PAMPS-filled nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes at different relative humidities: (a) 60 RH%, and (b) 80 RH%.

Vapor permeability and permselectivity

Vapor permeabilities of polymer membranes can be obtained by measuring the transfer rates of vapor molecules through membranes. In this work, four different vapors, i.e., water, acetone, methanol and DMMP, were chosen as permeants. Figure 5 shows typical water vapor transfer rate data of a linear PAMPS-filled nonwoven membrane. The weight of permeation cell was recorded until its loss reached the steady state and the weigh loss (0.038 g/day) was calculated from the slope of the weight–time curve shown in Figure 5. Based on the vapor transfer rate, the vapor permeability of the membrane can be obtained. Figure 6 and Table III show the permeabilities of four different vapors for linear PAMPS, linear PAMPS-filed nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes.

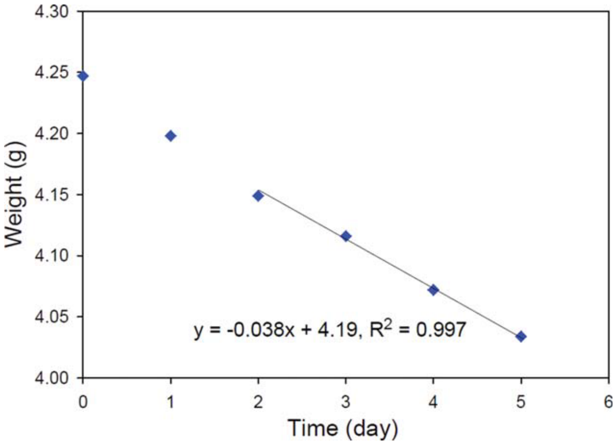

Figure 5 Typical water vapor transfer rate data of linear PAMPS-filled nonwoven membrane. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

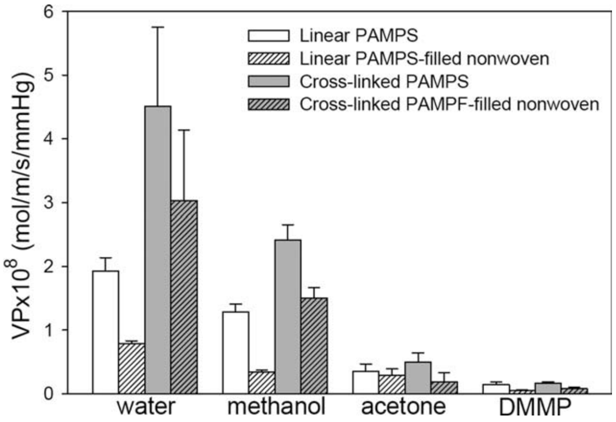

Figure 6 Vapor permeabilities of linear PAMPS, linear PAMPS-filed nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes. Vapors used are water, acetone, methanol, and DMMP.

The permeabilities of linear PAMPS and linear PAMPS-filled nonwoven membranes are lower than those of crosslinked ones in all four vapors. This is mainly caused by the introduction of microheterogeneity by the crosslinking of PAMPS.[27] From Figure 6, it is also seen that PAMPS-filled nonwoven membranes show lower vapor permeabilities than PAMPS membranes without nonwoven hosts. Nylon nonwovens consist of randomly deposited fibers, which produce tortuous pathways for vapor molecules to pass through and retard their penetration across the membranes. In addition, confining PAMPS in the open pores of nonwoven fabrics also suppresses the polymer swelling, and hence reduces the vapor permeation to a minimum. The significant lower membrane permeabilities by the addition of nylon nonwovens indicate the potential applications of polymer-filled nonwoven membranes for separating vapors.

Among the four vapors studied, DMMP is an important simulant of Sarin (GB), one of the organophosphorus nerve agents, due to their similarity in

TABLE III
Permeability of Linear PAMPS, Linear PAMPS-Filled Nonwoven, Crosslinked PAMPS, and Crosslinked PAMPS-Filled Nonwoven Membranes

Permeant membrane	Water	Methanol	Acetone	DMMP
Linear PAMPS	1.93 ± 0.21	1.28 ± 0.13	0.35 ± 0.12	0.14 ± 0.04
Linear PAMPS-filled nonwoven	0.78 ± 0.05	0.34 ± 0.04	0.30 ± 0.10	0.05 ± 0.006
Crosslinked PAMPS	4.51 ± 1.25	2.41 ± 0.25	0.50 ± 0.14	0.17 ± 0.02
Crosslinked PAMPS-filled nonwoven	3.03 ± 1.10	1.50 ± 0.16	0.19 ± 0.14	0.08 ± 0.03

molecular size, water-solubility, and chemical structure. As shown in Figure 6, the penetration of DMMP vapor across PAMPS membranes is substantially lower than that of water. This trend is strongly required for chemical protection membranes, which shield humans from hazardous chemical vapors while still transporting water vapor. To study the potential application of PAMPS-filled nonwoven membranes for chemical protection, the permselectivities on DMMP over water were measured for linear PAMPS, linear PAMPS-filed nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes and the results are shown in Figure 7. It is seen that crosslinked PAMPS-filled nonwoven membranes exhibit the highest permselectivity among all four membranes.

From Figure 7, it is also seen that, although the permselectivity of crosslinked PAMPS membranes is improved by the presence of nonwoven hosts, confining linear PAMPS in nylon nonwovens does not significantly change the membrane permselectivity. In addition to chemical protection, the permselective permeable membranes can be also used in water purification and separation of gas or vapor mixtures.

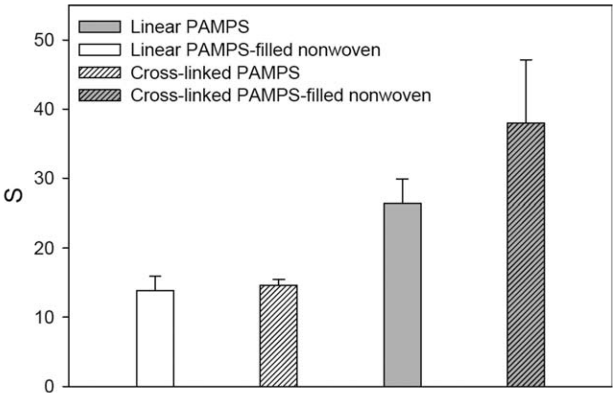

Figure 7 Permselectivities on DMMP over water for linear PAMPS, linear PAMPS-filed nonwoven, crosslinked PAMPS, and crosslinked PAMPS-filled nonwoven membranes.

CONCLUSIONS

Linear and crosslinked PAMPS membranes were synthesized by radical polymerization method using AIBN as an initiator and EGD as a crosslinker. PAMPS-filled nylon nonwoven membranes were prepared through completely filling the open pores in the nylon nonwoven fabric. PAMPS-filled nylon nonwoven membranes show better dimensional stability and mechanical properties than PAMPS membranes without nonwovens. The conductivities of PAMPS-filled nylon nonwoven membranes are lower than those of PAMPS membranes without nonwoven hosts but are still sufficient for many practical applications. PAMPS-filled nylon nonwoven membranes have lower permeability for water, methanol, acetone, and DMMP vapors than PAMPS membranes without nonwovens. The preferential permeation of water vapor compared to DMMP vapor suggests the potential application of PAMPS-filled nonwoven membranes as chemical protection membranes.

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