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Development of thin-film composite PRO membranes with high power density

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ABSTRACT

Pressure retarded osmosis (PRO) has recently attracted much attention as a promising alternative energy technology to overcome the problems posed by fossil fuel consumption and greenhouse gas emissions. PRO is a process of generating power using the energy potential that arises from the difference in concentration between two solutions separated by a selective semipermeable membrane. In order to commercialize PRO, new methods for producing high-performance PRO membranes must be developed. The power density of a PRO membrane, calculated as the product of osmotically induced water flux and the trans-membrane hydraulic pressure difference, is the key performance measure for these membranes. This study aims to improve membrane components and to develop a polyamide composite PRO membrane with a high power density. To this end, we determined the characteristic factors of a PRO membrane that contribute to increase the osmotically induced water flux and estimated their effect on the power density.

Keywords: Pressure retarded osmosis; Thin-film composite membrane; Power density

1. Introduction

There has been increasing interest in the development of alternative energy sources as part of a global effort to tackle the problems produced by fossil fuel consumption and greenhouse gas emissions. As part of this effort, energy-efficient methods for seawater desalination have been developed and extensively studied to lower the energy required to produce fresh water. Reverse osmosis (RO) requires substantially less energy compared to thermal processes and so has dominated the desalination market for decades [1–4]. Despite its wide use in seawater desalination, RO still has a number of unsolved technical problems. The RO membrane is susceptible to membrane fouling and so a preprocessing step is necessary to remove particulate matter and micro-organisms from inlet seawater.

It is also important to reduce the hardness of the water. In addition to this unavoidable preprocessing step, boron must be removed because the boron ions are not removed by RO at the same rate as other ions present in seawater [5–7]. Besides such mechanical processing steps, the most urgent challenge facing RO seawater desalination is to lower the energy consumption. Even though RO uses less energy than thermal distillation, it is still higher than conventional methods

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of water production. A new and revolutionary RO/pressure retarded osmosis (PRO) desalination system [8] developed to solve these energy usage problems, is considered the most promising technology. In PRO, power is generated from the energy potential resulting from the difference in concentration between two solutions separated by a semipermeable membrane. It shares the same underlying principle as forward osmosis, in which pure water and power can be obtained using a semipermeable membrane that separates a feed solution of low concentration and a draw solution of high concentration. The power generating potential is the salinity gradient energy that exists between two solutions with different salt concentrations. In PRO, freshwater and seawater are used as the low-concentration and high-concentration solutions, respectively. The preprocessed, low-concentration solution permeates into the high-concentration solution across the semipermeable membrane by osmosis, and the flux of water causes a turbine to rotate, thereby generating energy. In other words, the osmotic pressure arising from the concentration, difference between two solutions is converted into a form of hydraulic pressure, which can be used to turn a turbine and produce energy. Its power density, which is the product of osmotically induced water flux, is the key indicator of the performance of a PRO membrane and the pressure imposed on the high-concentration solution [9,10].

Potentially, the total global energy production from the mixing of seawater and freshwater in the PRO method could be up to two TW. Salinity gradient power has the potential to provide as much as 80% of current global power needs, and can be generated regardless of weather conditions. In contrast, solar and wind energy are highly dependent on climatic and seasonal conditions. Conventional PRO mixes seawater and river water, but an emerging trend is to utilize the extremely saline water produced as part of the desalination process, thus producing energy more efficiently. However, most desalination plants are coastal, and hence, other freshwater resources must be used. These include low-salinity surface water, wastewater treatment effluent, recycled water, transitional water, and groundwater. Using the power generated by PRO to run the seawater desalination plant could reduce the overall energy consumption of the desalination plant. Such a PRO system necessitates various new technologies to make the system viable, such as low-pressure seawater intake energy recovery devices and pre-processing to prepare the various types of feed water for use in PRO [11–14]. Perhaps the most important is the removal of micro-organisms from feed solutions. Efficient preprocessing is essential to minimize the fouling of the PRO membrane and to maintain their performance. High pressure is necessary to retard the osmotic flow from the highly concentrated brine and minimize the internal and external concentration polarization, so the membranes must be able to withstand these pressures. The development of new materials and methods is vital to improve production efficiency, reduce costs of freshwater production, and to make commercially viable a combined desalination-power generating plant using existing plants [15,16]. Ultimately, the key to commercializing PRO is the development of a high-performance membrane.

In this study, we identified the characteristic features of a PRO membrane associated with the osmotically induced water flux and estimated their effects on power density. Based on our findings, we improved the features associated with membrane structure and developed a polyamide composite PRO membrane that exhibited high power density.

2. Materials and methods

2.1. Preparation of a polysulfone backing layer

The PRO support membrane was prepared by the phase inversion precipitation method. The phase inversion precipitation method is one of the most commonly used methods of membrane production. Whereby, the polymer is precipitated by following the solution exchange of good solvent and non-solvent [16–18]. The most critical physico-chemical properties influencing the membrane structure in solvent exchange methods are the polymer precipitation rate and the good-solvent/non-solvent exchange rate. A high precipitation rate results in a macroporous finger-like structure, whereas a low precipitation rate yields a microporous sponge structure.

Preparation of a polysulfone (PSf) (Solvay) was used as polymer in preparation of the membrane casting solution. *N*,*N*-dimethylformamide (DMF, anhydrous, 99.8%) and *N*-methyl-2-pyrrolidone (NMP, anhydrous, 99.8%) were used as the polymer solvents without further purification. Deionized water was used as a coagulant. To prepare polyamide PRO membranes, PSf was dissolved in DMF and NMP by stirring at 40˚C and then stored in a desiccator for at least 24 h prior to casting. The membrane substrate was cast from polymer solutions onto a nonwoven and porous fabric backing layer. The cast membranes were immediately immersed into a deionized water coagulation bath for several minutes to ensure complete precipitation of the polymer had occurred. The membranes were then washed with DI water before the interfacial polymerization was carried out.

2.2. Preparation of a polyamide active layer

An interfacial polymerization reaction is used to produce a thin coat of polyamide on the support layer surface. The reaction occurs between aqueous 1,3-phenylenediamine (MPD, >99%) and 1,3,5-benzenetricarbonyl trichloride (TMC, 98%) dissolved in organic solvent. The prepared support layer is immersed in the aqueous MPD solution for about 1 min, followed by the removal of excess aqueous solution. The support layer is then immersed in the TMC solution for about 1 min, followed by the removal of excess amine solution. The active layer formed serves as a connection to the support layer and hardens the membrane allowing it to resist the high-pressures of the PRO process. Preparing a thin membrane of uniform density is critical to ensure optimal water flux and salt rejection. The reaction rate should be kept high using an adequate catalyst because an insufficient reaction rate causes a low flow rate and coarse crosslink formation due to increased active layer thickness.

2.3. Membranes performance testing in PRO mode

In PRO, the volume of the draw solution increases due to the osmotic flow of freshwater into the highly saline draw solution the PRO semipermeable membrane. The increased quantity of water of the draw solution increases the pressure in the draw solution chamber, and power is generated by water exiting the chamber and driving the turbine. The water flux across the PRO membrane is defined by Eq. (1):

$$J_w = A(\Delta\pi - \Delta P) \tag{1}$$

where J_w is the water flux, A is the water permeability coefficient of the membrane, $\Delta\pi$ is the transmembrane osmotic pressure difference, and ΔP is the transmembrane hydraulic pressure difference. Power generated by the transmembrane osmotic pressure difference in PRO is expressed as power density (W), which is obtained by the product of the water flux (J_w) and the hydraulic pressure difference across the membrane (ΔP) imposed on the membrane from the side of the draw solution [19–21].

$$W = J_w\Delta P = A(\Delta\pi - \Delta P)\Delta P \tag{2}$$

By differentiating Eq. (2) with respect to ΔP, it is found that the maximum power density ($W_{\max}$) can be obtained at $\Delta P = \Delta\pi/2$.

$$W_{\max} = \frac{A\left(\Delta\pi - \frac{\Delta\pi}{2}\right)\Delta\pi}{2} = \frac{A\Delta\pi^2}{4} \tag{3}$$

Seawater contains, on average, 35 g/L NaCl (0.6 M) and its ion concentration is 1.2 M. Applying the van't Hoff equation, the osmotic pressure of seawater is calculated to be 28.7 bar at 20℃. Therefore, the theoretical maximum power density for this concentration is obtained at a hydraulic pressure difference of 14 bar. When using brine containing 70 g/L of NaCl as the draw solution, the osmotic pressure is 56 bar; thus, the maximum power density can be achieved at a hydraulic pressure difference of 28 bar. However, this is an ideal value that is calculated based on the following assumptions: (i) DI water is used as the feed solution, (ii) there is no salt co-flux across the membrane, and (iii) there is no loss in osmotic pressure.

The power density of a PRO membrane was experimentally measured using 35 g/L NaCl solution as the draw solution (simulating seawater), and DI water as the feed solution. The performance of the PRO membrane was tested using a flat-sheet membrane cell with an effective membrane area of 6 cm × 11 cm at a flow rate of 500 mL/min for the feed and draw solutions. The osmotically induced water flux was measured by monitoring the change in the weight of the feed and draw solutions. In order to prevent the accuracy of performance evaluation being affected by dilution of the draw solution, the volumes of feed and draw solutions were set at 2 and 5 L, respectively. By doing this the dilution of the draw solution was limited to less than 5%, minimizing its effect on the water flux. The power density was then calculated by multiplying the measure water flux by the imposed pressure.

2.4. Measurement of characteristic factors of the PRO membrane

Water flow across a PRO membrane is induced by the transmembrane osmotic pressure difference ($\Delta\pi$). Since the pressure imposed on the PRO membrane is exerted in a direction opposite to the water flow, the water flux (J_w) becomes smaller with an increase in the pressure difference (ΔP). However, in order to estimate the actual water flux, three factors should be considered: (i) the loss in osmotic pressure due to salt co-flux, (ii) the decrease in the effective osmotic pressure due to impeded salt diffusion within the support layer, and (iii) the dilution of the draw solution in the PRO membrane active layer due to the permeation of feed water into the draw solution. These three factors influencing water flux can be described by the salt permeability constant (B), the resistance to salt, $K_R = S/D$ (where D = salt diffusion coefficient and S is a factor related to the support structure), and J_w/k, which denotes the relationship between the water flux and mass transfer in the draw solution (k is the mass transfer coefficient) [15,16,22]. When the water flux (J_w) is defined by taking into account these factors, Eq. (1) can be corrected to give Eq. (4):

$$J_w = \frac{1}{K_R}\ln\left(\frac{A\pi_{D,m} + B - J_w - A\Delta P}{A\pi_{feed} + B}\right) \tag{4}$$

S can be reduced by developing a highly porous membrane that allows more direct mass transfer and is sufficiently thin to reduce internal concentration polarization, so that higher power densities can be achieved. The values of the characteristic factors A and B can be measured in RO mode. Salt rejection and water flux can be measured at 10 bar with the same PRO measurement equipment using 1,000 ppm NaCl solution instead of the draw solution [23,24]. The values of A and B are determined with Eqs. (5) and (6) [25,26].

$$J_w = A(\Delta\pi - \Delta P) \tag{5}$$

$$B = \frac{J_w(1-R)}{R}\exp\left(-\frac{J_w}{K}\right) \tag{6}$$

3. Results and discussion

3.1. Characterization of PRO membranes

The support layer plays a pivotal role in enhancing the water permeability coefficient of a PRO membrane. The optimal structural parameters of the support layer are known to be thinness, low tortuosity, and high porosity, all of which are conducive to lowering the internal concentration polarization, thus enhancing the water permeability coefficient by osmosis [27]. Although a finger-like structure is more suitable for enhancing porosity, we opted for a pressure-resistive sponge-type structure for the membrane to be used in PRO operations at 25 bar. Fig. 1 depicts a cross-sectional scanning electron microscope (SEM) image of the PRO membrane. The support layers were prepared using the previously discussed method at varying polymer concentrations. In PRO, because water flows from the support layer to the active layer by osmosis the support layer must be strongly bonded to the nonwoven fabric, in contrast to the flow in RO [27,28]. The hydrophilicity of the PRO support layer is important, as the transmembrane water flow is driven by osmosis, in contrast, the high pressures in RO mean that the hydrophilicity of the support layer is relatively unimportant.

3.2. Power density measurement on a PRO flat-sheet membrane

Fig. 2 shows the flux rate over the PRO membrane at different pressures of NaCl, which draw solution with concentrations of 35 and 70 g/L (simulated seawater and brine, respectively). As expected, the flow rate decreased in inverse proportion to ΔP for both NaCl solutions. It is worth mentioning that the osmotic pressure of 35 g/L NaCl solution is 27.6 kgf/cm^2, while that

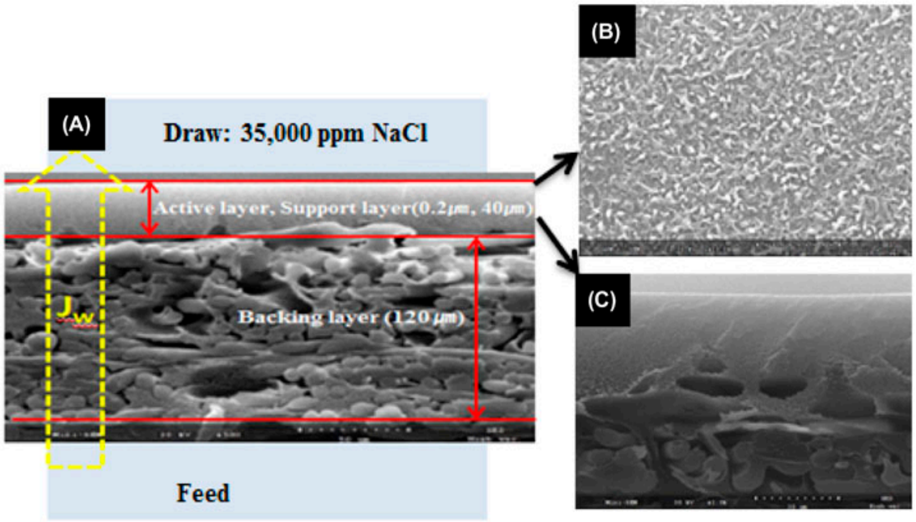


Fig. 1. SEM images of TFC-PRO membrane with backing layer: (A) cross section, (B) magnified view of the polyamide active layer surface, and (C) magnified view of the skin layer at the top of the PSf porous support with dense, sponge-like morphology.

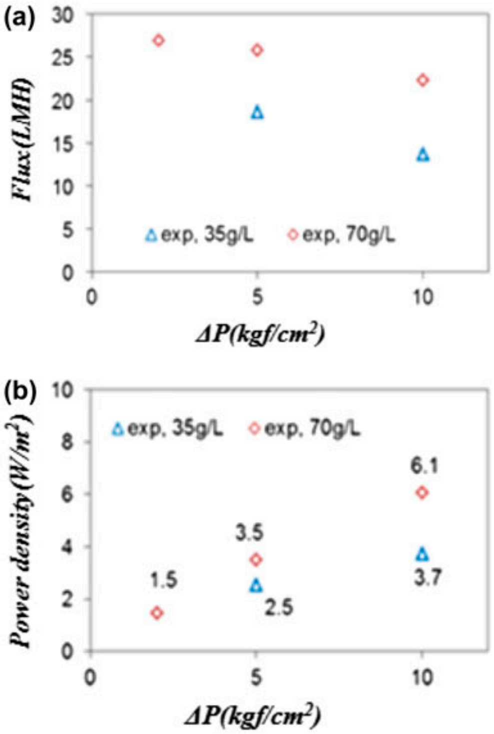


Fig. 2. Effect of draw solution concentration on: (a) water permeability and (b) power density.

of 70 g/L NaCl solution is 55.3 kgf/cm^2. Osmotic pressure increases with solute concentration. Therefore, the increase in concentration of the draw solution leads to an increase in water flux under the same operating pressure. Fig. 2(a) shows that the water flux was higher when the draw solution concentration increased from 35 to 70 g/L. At an operating pressure of 10 kgf/cm^2, power density.

When seawater and brine were used as the feed solution 3.7 and 6.1 W/m^2 were obtained, respectively. Fig. 2(b) shows that a higher power density is achieved at higher pressure in brine solutions. Examining the water flux at different draw solution concentrations at the same operating pressure shows that relationship between power density and draw solution concentration is not exactly proportional. For example, at an operating pressure of 5 kgf/cm^2, $\Delta\pi$, the driving force for osmotic water flow, increased 2.2-fold from 22.6 to 50.3 kgf/cm^2 as the concentration increased from 35 to 70 g/L NaCl (Table 1). The water flux should also increase proportionally (Eq. (1)), but was in fact limited to a 1.4-fold increase. Although the water flux is supposed to increase 2.2-fold as per Eq. (1), the actual increase was limited to 1.4-fold. A similar tendency was also exhibited under an operating pressure of 10 kgf/cm^2 (Table 2).

Table 1
Comparison between $\Delta\pi$ and ΔP and the water permeability at an operating pressure of 5 kgf/cm^2

NaCl concentration (g/L)	35 g/L	70 g/L
$\Delta\pi$–ΔP (kgf/cm^2)	22.6	50.3
(LMH)	8.6	25.8
Ratio of $\Delta\pi$–ΔP	1	2.2
Ratio of water permeability	1	1.4

Table 2
Comparison between $\Delta\pi$ and ΔP and the water permeability at an operating pressure of 10 kgf/cm^2

NaCl concentration (g/L)	35 g/L	70 g/L
$\Delta\pi$–ΔP (kgf/cm^2)	17.6	45.3
Water permeability (LMH)	13.8	22.3
Ratio of $\Delta\pi$–ΔP	1	2.6
Ratio of water permeability	1	1.6

The reasons for this discrepancy between concentration and flux rate at a given pressure when applying Eq. (1) are the three aforementioned factors: (i) loss in osmotic pressure due to salt co-flux, (ii) decrease in the effective osmotic pressure due to impeded salt diffusion within the support layer, and (iii) dilution of the draw solution in the PRO membrane active layer due to the permeation of feed water into the draw solution. Eqs. (4)–(6) can be used to determine the values of S at a concentration of 35 g/L NaCl and at 10 kgf/cm^2 operating pressure using the salt rejection and water flux in 1,000 mg/L NaCl as well as osmotically induced water flux across the PRO membrane. The values of the characteristic factors of the PRO membrane are tabulated in Table 3.

3.3. Estimation of the PRO flat-sheet membrane power density

In PRO, the dependence of water flux on draw solution concentration and operating pressure can be estimated with Eq. (4), and the values of the characteristic factors are shown in Table 3. Fig. 3 presents the values of water flux and corresponding power density estimated under the operating pressure tested

Table 3
Values of the characteristic factors of the PRO membrane

A (L/m^2/h/kgf/cm^2)	B (L/m^2/h)	S (mm)
2.57	0.627	1.12

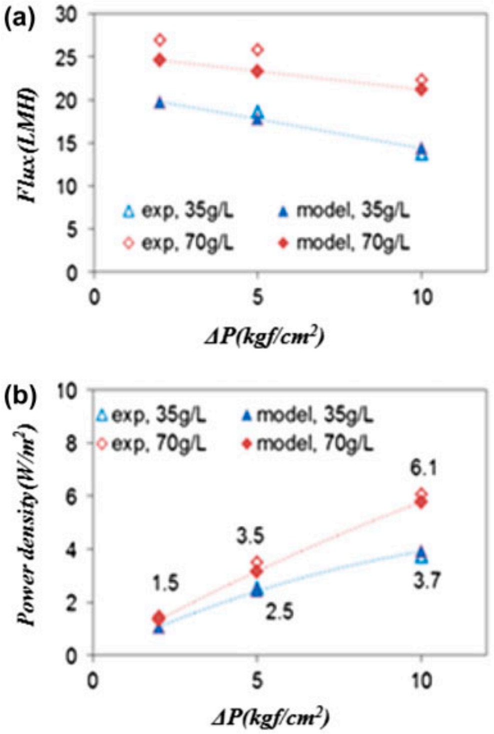


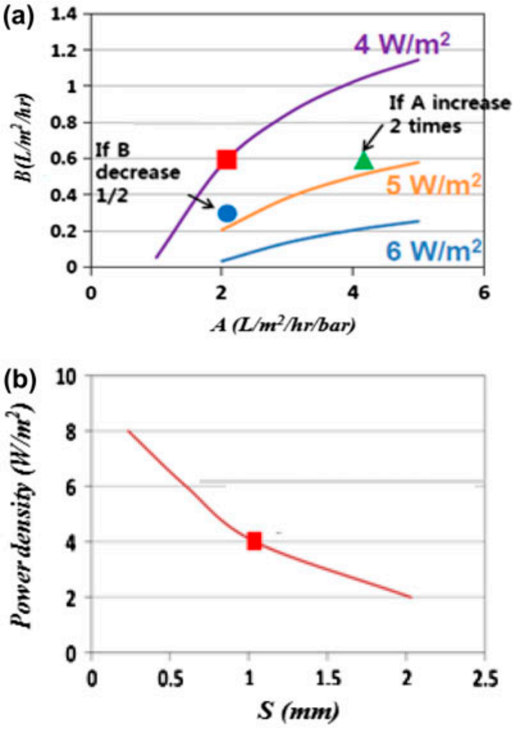
Fig. 3. Values of: (a) water flux and (b) power density estimated under the operating pressure tested for the two different NaCl solutions (35 and 70 g/L).

for the two different NaCl solutions (35 and 70 g/L). As shown in Fig. 3, the values estimated using Eq. (2) were similar to the experimentally measured values. Eq. (2) can also be used for estimating the maximum power density when a 70 g/L NaCl draw solution is used. The effective osmotic pressure ($\Delta\pi_{\text{eff}}$) generated across the PRO membrane can be expressed by the equation $\Delta\pi_{\text{eff}} = \Delta\pi/(B \cdot K_{\text{R}} + 1)$ including the salt permeability constant (B), and the resistance to salt diffusion (K_{R}) inside the membrane. The effective osmotic pressure imposed by a 70 g/L NaCl draw solution is 52 kgf/cm^2 and the maximum power density with these conditions occurs at a hydraulic pressure difference of 26 kgf/cm^2, given that the maximum power density occurs at half the effective osmotic pressure. Therefore, the maximum power density that can be produced by a 70 g/L NaCl draw solution is estimated to be 10.6 W/m^2 at a transmembrane hydraulic pressure difference of 26 kgf/cm^2.

3.4. Approaches to improving the performance of a flat-sheet PRO membrane

These results show that the performance of the PRO membrane is affected by the intrinsic membrane parameters A, B, and S. Therefore, to increase membrane performance, the membranes must have increased water permeability, decreased the salt permeability and improved membrane structure (porosity and tortuosity). The maximum osmotic pressure can be estimated from concentration of the feed and draw solutions. In order to obtain the maximum water flux from the osmotic pressure difference thus determined, the membrane must have a high water permeability coefficient (A). B and K_{R} are factors that are difficult to control because unlike an ideal semipermeable membrane, through which water passes and salts do not pass, PRO membranes usually incur transmembrane salt co-flux thus failing to reach the ideal osmotic pressure. A large S value means a low salt diffusivity, leading to an osmotic pressure lower than the ideal value. Therefore, the best strategy to obtain the maximum water flux is to maximize A and minimize B and S.

Using Eq. (4), we have estimated the effect of each of the characteristic membrane factors on the power density, as shown in Fig. 4. The results reveal that the power density can be improved from 4 to 5 W/m^2 if A is doubled, or if B is halved, at constant S



*NaCl 35g/L, 10 bar

Fig. 4. Prediction of PRO performance based on A, B, and S value: (a) effect of A and B at constant S and (b) effect on power density of S.

Table 4
Effect of different backing layers on PRO performance

	Nonwoven fabric			Porous fabric		
	A	*B*	*C*	*D*	*E*	*F*
Air permeability (cc/m^2/sec)	4.8	9.0	21.4	67	127	261
Thickness (μm)	124	118	77	93	74	74
Power density (W/m^2)	3.7	3.4	4.0	3.6	7.7	5.2

(Fig. 4(a)). In contrast, halving S, at constant A and B, leads to an increase from 4 to 6 W/m^2 (Fig. 4(b)). Therefore, we directed our efforts to finding a method to reduce the S values.

3.5. Enhancement of power density via improvement of PRO support layer

S can be reduced either by modifying the polymer support layer or the nonwoven fabric backing layer, for example, by increasing the porosity and decreasing the thickness and tortuosity of the polymer support layer, or using a thin porous nonwoven fabric backing layer. Table 4 summarizes the efforts to modify the backing layer using various types of thin and porous nonwoven fabric. Contrary to expectation, no significant improvement in power density was achieved using thin and porous fabrics. The support layer was then modified, using various porous fabrics. The permeability of the nonwoven fabrics and the porous fabrics was measured. The porous fabrics are over 10 times more porous than the nonwoven fabric, thus exerting less resistance to transmembrane flow. Of the porous fabrics tested, Type *B* yielded a twofold improvement in power density over nonwoven fabric PRO membrane.

4. Conclusions

In this study, we analyzed the effects of the characteristic factors (A, B, and S) of a PRO membrane on its power density. The results revealed that S exerts a great influence on the PRO performance. S was substantially reduced when the backing layer was replaced with a thin and porous fabric. Using this material we developed a PRO membrane that exhibited a twofold improvement in power density from 3.7 to 7.7 W/m^2 when operated using seawater concentrations.

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