Materials Today Communications 24 (2020) 101104

Contents lists available at ScienceDirect

Materials Today Communications

journal homepage: www.elsevier.com/locate/mtcomm

Development of high-permeable PSf/PANI-PAMPSA composite membranes with superior rejection performance

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ARTICLE INFO

Keywords:
Polyacid doped PANI
Polysulfone
Ultrafiltration
Hydrophilic membrane
High permeability

ABSTRACT

Polysulfone is the most popular polymeric matrix material for fabrication of ultrafiltration membrane but suffers from its hydrophobic nature that caused low flux and fouling problems that limits its development and industrial applications. The blending PSf with hydrophilic polymers is an attractive and effective technique to improve the hydrophilicity of membrane. Here poly(2-acrylamido-2-methyl-1-propane sulfonic acid doped polyaniline was synthesized and used as a hydrophilic additive in polysulfone to obtain ultrafiltration range membranes with higher flux. Infrared results supported the increased hydrophilicity of the membranes with PANI-PAMPSA additive which is confirmed by dynamic contact angle analysis results as well. The cross-sectional SEM images revealed that the pristine and blend membranes were asymmetric structured and had dense skin layer, finger-like pores on the top and sponge-like pores underneath the porous support layer. Also, the skin layer of the membrane became thinner and denser with the addition of 0.1 and 0.25 % and thicker with the addition of 0.5 additive ratio. The initial contact angles of P0, P1, P2 and P3 measured as 85, 81, 79 and 72°, respectively. Pure water permeability of the P0, P1, P2 and P3 membranes calculated as 14.0, 17.2, 23.4 and 21.3 $L.m^{-2}.h^{-1}.bar^{-1}$, respectively. The rejection ratios of the prepared membranes increased from 90.1 (P0) to 95.8 and 98.4 % for P1 and P2 membranes then decreased to 95.2 % for P3 membrane. The rejection results indicated that the prepared membranes can be classified as ultrafiltration membranes with molecular weight cut-offs (MWCOs) lower than 66 kDa g mol^{-1}.

1. Introduction

Polysulfone is the most popular polymeric membrane matrix material for fabrication of ultrafiltration (UF) membrane, due to its good physical and chemical properties with its excellent film-forming capability [1]. However, PSf membranes usually have low flux problem and also suffers from serious fouling problem because of the hydrophobic structure [2], that limits its development and industrial applications also increase its investment and operating costs [3]. Increasing the hydrophilicity of the membrane have been considered to control low flux and fouling problem [4]. The blending of hydrophilic polymer and PSf is an attractive and effective technique to obtain improved performance membranes [5,6], due to the process is less complicated and inexpensive [7].

Polyaniline (PANI) is one of the most important polymers for membrane separation processes both as base membrane material and additive in membrane structures. It have had many applications like ultrafiltration [8], ultrafiltration [9], gas separation [10], pervaporation [11] membranes and is well studied due to its processibility, environmental stability [12], outstanding physical properties [13], conductivity [14–16] and simple synthesis from a relatively cheap monomer [16,17]. However, the application of PANI is restricted by the relatively poor mechanical properties [16,18–20] and also easy leaching out the small dopants like HCl and dedoping from PANI by aging or by contact with water or solvents [21,22]. On the other hand, it is believed that the HCl dopant species are so small that they evaporate or are sublimed out of the polymer [22]. The loss of dopants will change the structural and morphological characteristics as well as a loss of electrical conductivity of the polymer [23]. To overcome this possible leaching the dopant, the use of polymeric acids can be an effective way because of the large dopants can be strongly bound to the polymer with the strong interaction between the acidic groups and the steric effect of the larger size of acids [24]. So polymer acid doped PANI more environmentally stable than small acid doped PANI like HCl [25]. Among the different polyacids, Poly(2-acrylamido-2-methyl-1-propane sulfonic acid) (PAMPSA) is a suitable polyacid dopant with sulfonic groups that has good interactions with the polyaniline backbone. On the other hand, PANI-PAPMSA has good conductivity [25]. Good conductivity gave membran conductive properties and high conductivity increases surface hydrophilicity. Also conductive membranes can offer

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https://doi.org/10.1016/j.mtcomm.2020.101104
Received 25 December 2019; Received in revised form 26 March 2020; Accepted 27 March 2020
Available online 03 April 2020

innovative solutions for membrane-fouling control while maintaining enhanced filtration performance [26] and also the polar structure of PANI-PAMPSA provides hydrophilic character to the membrane [27,28]. These characteristics make PANI-PAMPSA a promising alternative blending material to obtain hydrophilic and high-permeance PSf membranes. Although there are a some studies about PANI doped PSf membranes, in this study, PANI-PAMPSA (polyacid doped PANI) used as an additive. The PANI-PAMPSA is a novel additive for PSf membranes and as I know there is no study about PANI-PAMPSA modified PSf membranes in literature. Therefore in this study, PANI-PAPMSA has been synthesized by using oxidative polymerization method and blended with PSf in different ratios. Then the PSf/PANI-PAMPSA blend membranes have been prepared by phase inversion technique. The chemical, morphological and surface properties were investigated and also permeability and rejection performances evaluated by using different methods.

2. Experimental

2.1. Materials

For the synthesis of the PANI-PAMPSA additive polymer, aniline, poly(2-acrylamido-2-methyl-1-propanesulfonic acid), ammonium persulfate ($(NH_4)_2S_2O_8$) were used. Polysulfone (PSf, Mw: 60,000), polyethylene glycol 1500 (PEG1500), N-methyl-2-pyrrolidone (NMP) used for the preparation of membranes. Also bovine serum albumin (BSA, Mw ~66 kDa) were used for membrane rejection performance and MWCO determination. All experiments performed using deionized (DI).

2.2. Preparation of PSf/PANI-PAMPSA blend membranes

Before the composite membrane preparation step firstly, the PANI-PAMPSA additive polymer was synthesized. Oxidative polymerization technique that used our previous study [29] was used for this synthesis. Synthesised PANI-PAMPSA polymer powder washed and filtered deionised water and acetone (5 times for each solvent) to remove unreacted chemicals, co-products and PANI oligomers. The chemical structure of the synthesized polymer was given in Fig. 1.

Table 1
Casting solution compositions of PSf/PANI-PAMPSA composite membranes.

Membrane	Casting solution (wt %)			
	PSf	PEG1500	NMP	PANI-PAMPSA
P0	18	2	80	0
P1	18	2	80	0.1
P2	18	2	80	0.25
P3	18	2	80	0.5

For the preparation of the casting solution for pristine membrane, PSf was completely dissolved in NMP, then pore former PEG1500 were added into the PSf solution and stirred for 12 h at room temperature by using magnetic stirrer. The percentages of the PSf:PF:NMP were adjusted as 18:2:80 in the pristine PSf membrane casting solution. For the preparation, the casting solution of the PANI-PAMPSA blended PSf membranes was similar to the preparation of the pristine PSf membranes. The only difference was the appropriate amount of PANI-PAMPSA added after dissolving the PSf and PF in NMP then stirred 12 h to obtain a homogeneous solution. Casting solution compositions of the PANI-PAMPSA blended PSf prepared membranes were given in Table 1.

After the preparation of the casting solutions, they left for 2 h for degassing. Then the phase inversion performed with immersion precipitation technique for the preparation of pristine and composite PSf membranes. The prepared membranes designated P1, P2, P2 and P3 that the compositions were given in Table 1.

2.3. Characterization of the membranes

Chemical compositions of the prepared composite membranes investigated Fourier transform infrared (FTIR) spectrometer. The surface and cross-sectional properties of the prepared membranes were studied by using a scanning electron microscopy (SEM). The surface hydrophilicity of the membranes investigated by using dynamic contact angle analysis. The filtration tests of prepared membranes were performed by

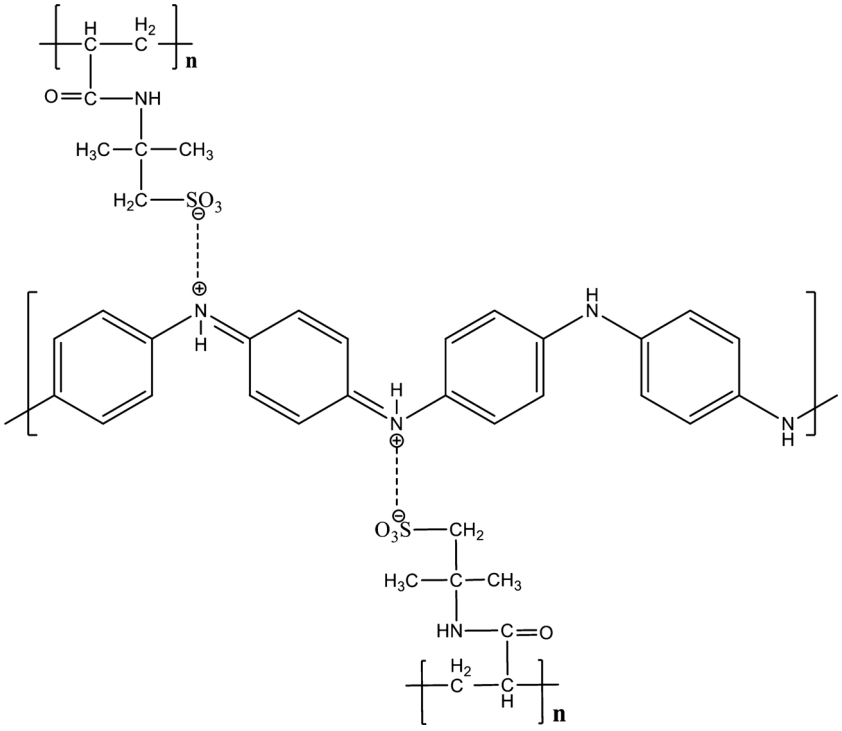

Fig. 1. PANI-PAMPSA polymer additive structure.

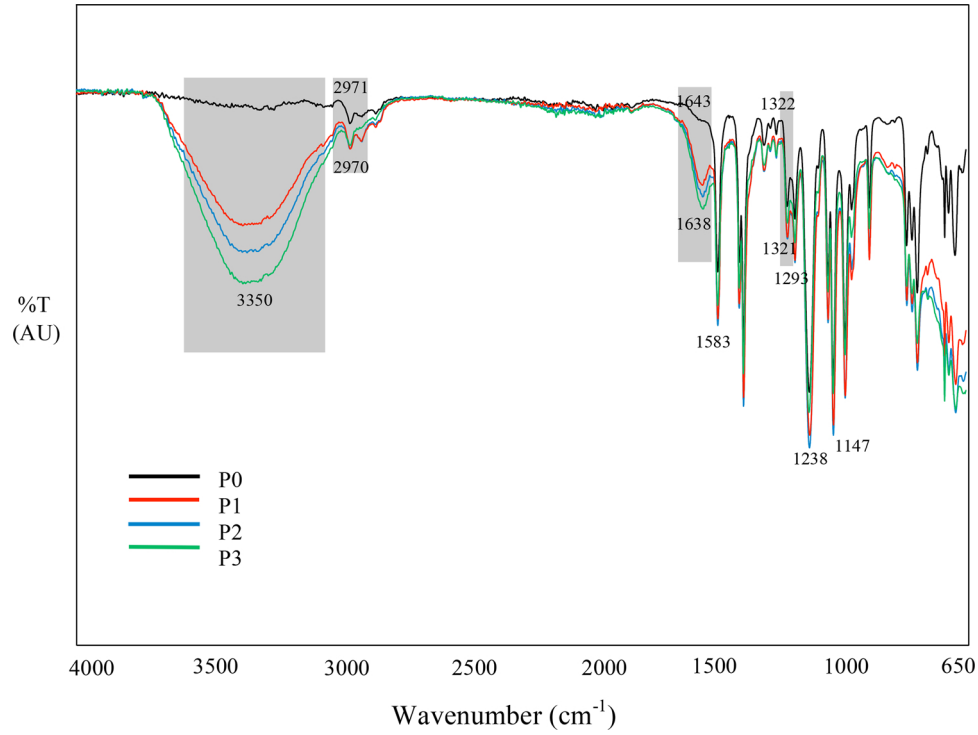

Fig. 2. FTIR spectra of the prepared membranes.

using a dead-end stirred filtration cell. Permeance and rejection studies were performed at 5 bar working pressure for all membranes. Permeance of the prepared membranes calculated by using Eq. 1. In this equation, Q, A and p means to permeate flow, membrane active area, and pressure, respectively.

$$P = \frac{Q}{A x p} \quad (1)$$

Rejection ratios were determined with the use of Eq. 2. In ti equation Cp and Cf refers to permeate and feed concentrations, respectively

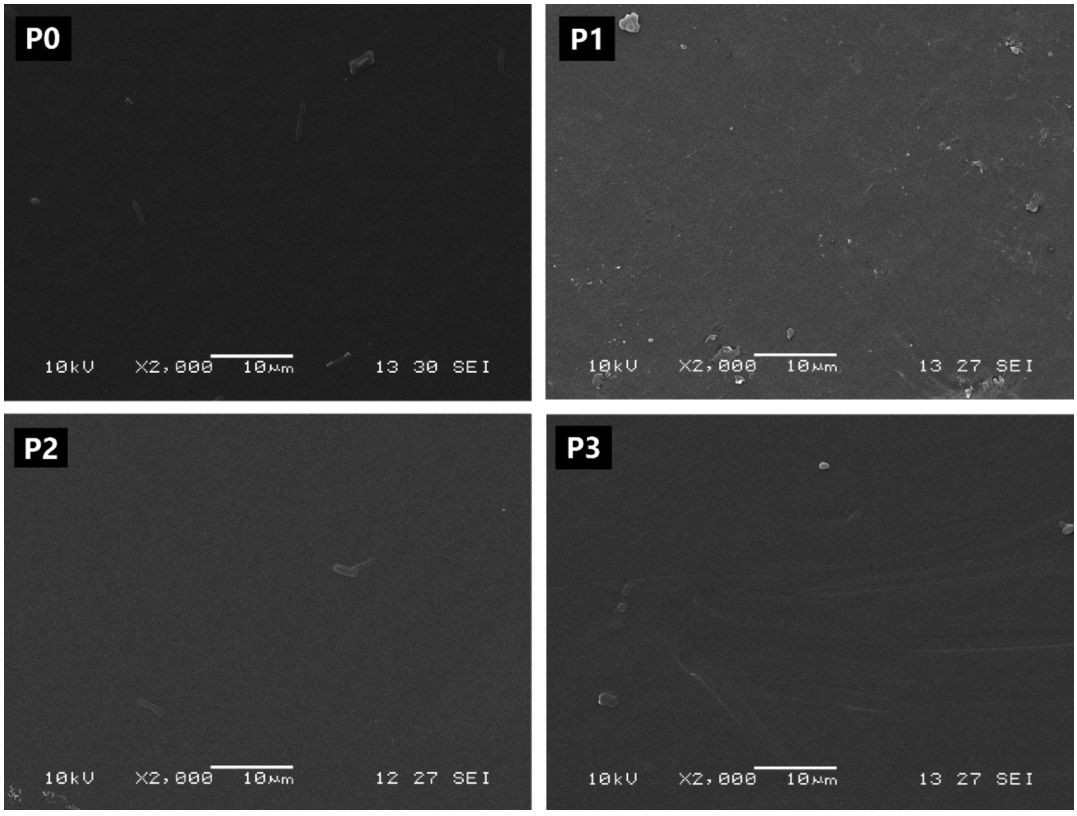

Fig. 3. Surface SEM images composite membranes.

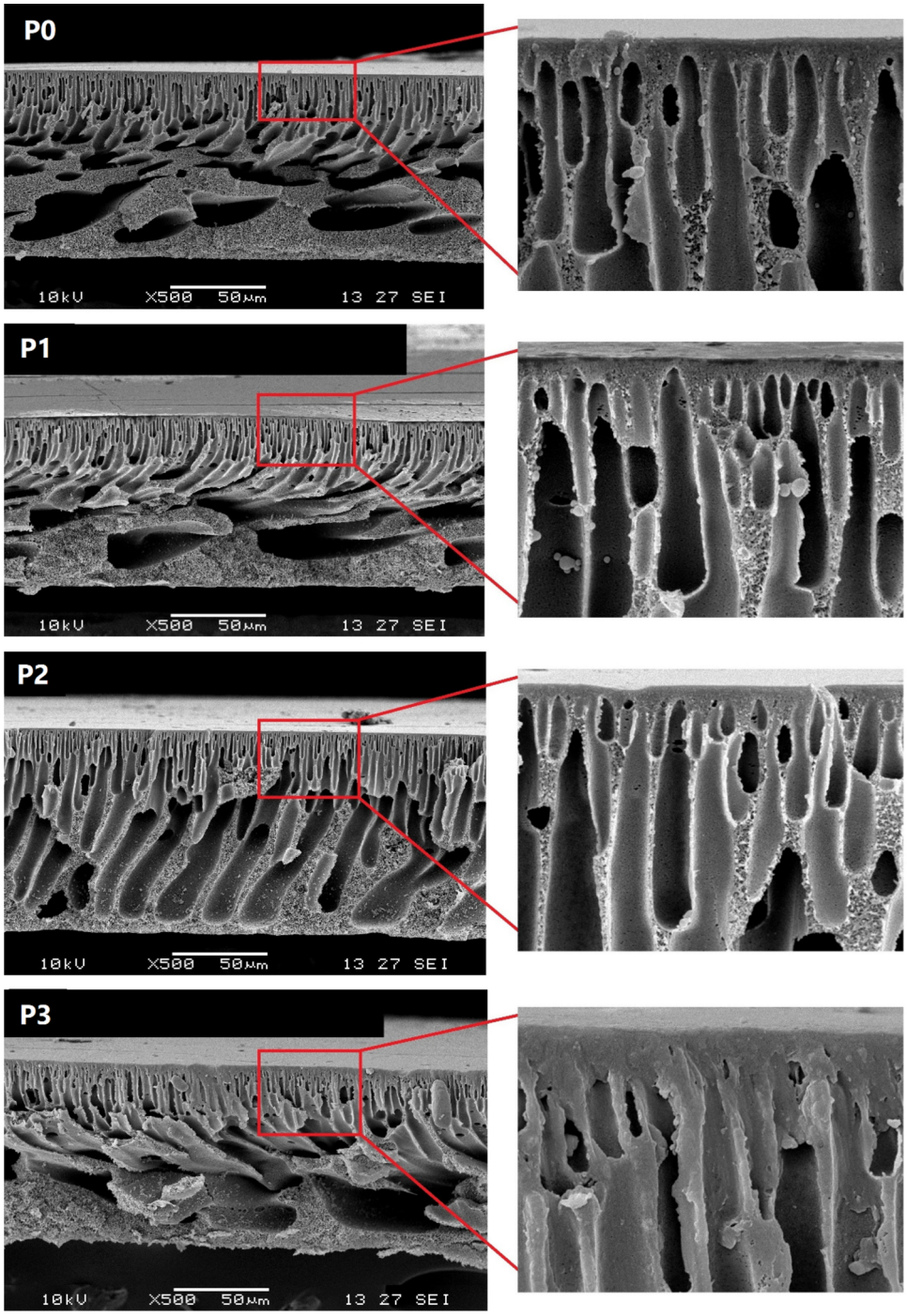

Fig. 4. Cross-section SEM images of composite membranes (x500 and x5000 magnifications).

$$R\,(\%) = \left(1 - \frac{Cp}{Cf}\right)x100 \quad (2)$$

The concentrations of BSA were determined by UV–vis. Spectrometer (Shimadzu Uv-2401) at λ_{max} = 280 nm wavelength, for the rejection studies.

3. Results and discussion

3.1. Infrared spectroscopy

The infrared spectra of the pristine and blend PSf membranes were given in Fig. 2. Typical bands of the pristine PSf were observed at 1147 and 1293 cm^{-1} (characteristic vibrations of O═S═O groups) [30], 2971 cm^{-1} (C–H stretching vibrations), 1585 cm^{-1} (conjugated C═C stretching vibrations of the aromatic ring) [31], 1322 cm^{-1} (C–SO_2–C asymmetric vibrations) [32]. The bands about 3350 and 1641 cm^{-1}, indicating OH stretching and bending frequencies, became apparent with the addition and increasing ratio of the PANI-PAMPSA. The result of OH stretching and bending bands shows that water adsorption is increased with the PANI-PAMPSA additive, that is contributed by the strong hydrophilicity of the membrane [33]. Also, small shifts of the positions of some peaks were observed with the addition of PANI-PAMPSA that can be seen on spectra: the band at 2971 shifted to 2970 cm^{-1}, the band at 1641 cm^{-1} shifted to 1638 cm^{-1} and also 1322 cm^{-1} shifted to 1321 cm^{-1}. These small vibration frequency changes attributed to

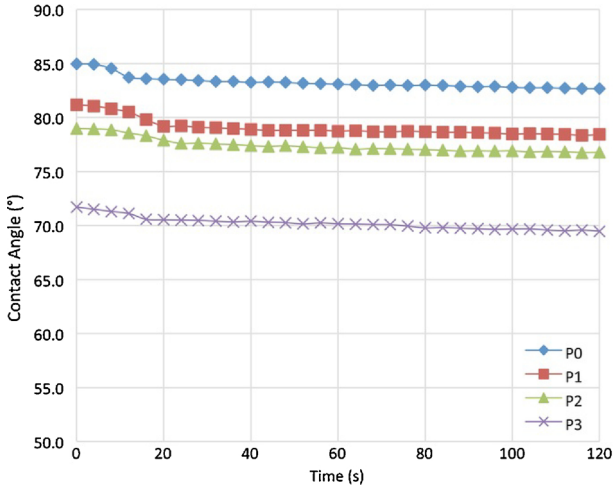

Fig. 5. Dynamic contact angle results of the prepared membranes.

interactions between PSf and PANI-PAMPSA additive.

3.2. SEM

The surface and cross-section SEM images of prepared composite membranes were given in Figs. 3 and 4. Surface SEM images showed that all the pristine and blend membranes have very similar surface images. The surfaces of the membranes are quite smooth and with the increasing of the PANI-PAMPSA additive became slightly smoother. Unlike surface images, the cross-section images showed some differences. The cross-sectional images reveal showed that all the composite membranes were asymmetric structured with a dense skin layer, finger-like pores on the top and sponge-like pores underneath the porous support layer. The most obvious differences between the pristine and blend membranes were increase on the length of the finger-like pores and changes the skin layer thicknesses with the increasing additive ratio. The pristine PSf membrane has a sponge-like structure more than finger-like pores but with the addition of 0.1 % additive the length of the finger-like pores started to increase then with the addition of the %0.25 PANI-PAMPSA almost all throughout the cross-sectional structure of membrane turned into finger-like pores that attributed to instantaneous demixing [34] due to the hydrophilic structure of the additive and therefore good interactions between solvent and additive. Unlike the low additive ratios, with the addition of 0.5 % PANI-PAMPSA, the length of the finger-like pores decreased and macro void formations observed beneath the short finger-like pores that can be seen on SEM image (d). Also, the skin layer of the membrane became thinner and denser with the addition of 0.1 and 0.25 % and thicker with the addition of 0.5 additive ratio. The skin layer thicknesses were calculated by using the average of the 10 measurements. Obtained thicknesses were as follows: 1.12, 0.96, 0.78 and 2.26 μm for P0, P1, P2 and P3 membranes, respectively. These changes son the thickness can be explained as follows: The phase separation process accelerated for P1 and P2 membranes due to the stronger interactions between non-solvent (water) and PANI-PAMPSA. Therefore the additive influenced the penetration rate of the water and increased the demixing rate of the casting solutions and as aforementioned the skin thicknesses of P1 and P decreased [35]. Due to the strong interactions solvent and additive, the outflow of the solvent delayed so that the phase inversion process would proceed comparatively slower demixing (delayed demixing) approach. In a nutshell, it can be easily seen on the cross-section structure of the 0.5 % blend membrane that the finger-like structures damaged, all the bulk structure became denser and there was some agglomeration on the bulk structure of the membrane.

3.3. Contact angle

To investigate the surface hydrophilicity of the prepared composite membranes, dynamic contact angle analysis was performed. The contact angle results in 120 s for pristine and blend membranes were given in Fig. 5. The initial water contact angles of P0, P1, P2 and P3 measured as 85, 81, 79 and 72°, respectively, then in 120 s they decreased to 83, 78, 77 and 70°. The decreases on the contact angle with the addition and increasing ratio of the PANI-PAMPSA indicated the increase on hydrophilicity of the membranes. Also, it can be easily seen that the contact angle decrease rates of the membranes were quite similar and contact angle differences between 0 and 120 s about 3° for all membranes. The contact angles for all membranes became stable about 20 s. The short contact angle stabilization times and the small differences between initial and terminal contact angles attributed to the smooth membrane surface with substantially free of nanopores.

3.4. Permeance and rejection tests

Membrane permeance was evaluated using pure water at 5 bar pressure after 7 bar compaction processes. Pure water permeance values obtained for the prepared membranes in 4000 s. were given in Fig. 6. The fluxes of the prepared membranes got a steady state permeance about in an hour. PWP of the P0, P1, P2 and P3 membranes calculated as 14.0, 17.2, 23.4 and 21.3 $L.m^{-2}.h^{-1}.bar^{-1}$, respectively. The results indicated that, PANI-PAMPSA additive had permeance enhancing effect of the PSf membrane for 0.1 and 0.25 % additive ratios. However, 0.5 % additive ratio caused a decrease on permeance, it was still higher than pristine PSf membrane's permeability.

The increase in permeability of the P1 and P2 membrane attributed to longer finger-like pore structure on the cross-section of the membranes and the higher hydrophilic character of the membrane owing to the hydrophilic additive. On the other hand, the P3 membrane exhibited a permeance decline that most probably caused by the thicker dense skin layer of the membrane and also pore-blocking phenomena due to the relatively higher ratio additive [36]. This approach on the flux decline supported by the cross-section SEM image of P3 membrane as well.

Similar to the permeability results, as shown in Fig. 7. The rejection ratios of the prepared membranes initially increased with the increasing additive ratio in the membrane and then decreased. The rejection ratios of the prepared membranes increased from 90.1 (P0) to 95.8 and 98.4 % for P1 and P2 membranes then decreased to 95.2 % for P3 membrane. The increase on rejection of the P1 and P2 attributed to the formation of membrane structure with smaller pores (especially skin

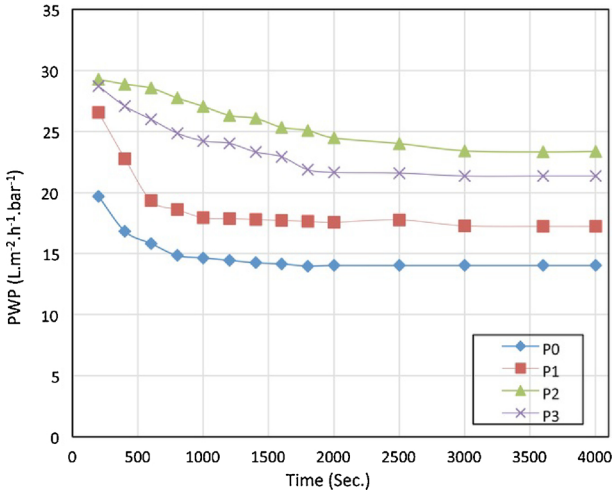

Fig. 6. PWP values of the PANI-PAMPSA blended PSf membranes.

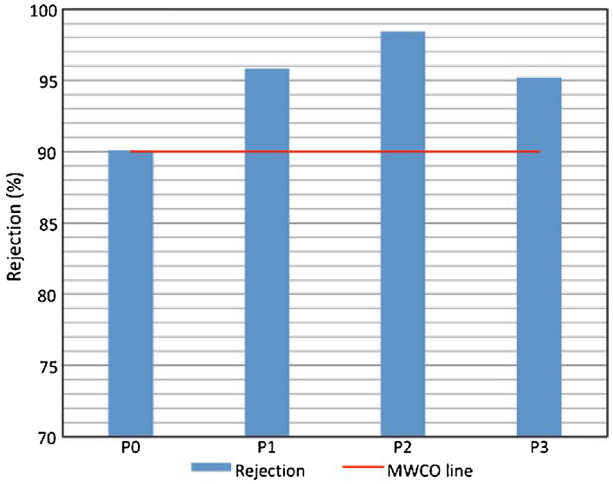

Fig. 7. Rejection ratios of the PANI-PAMPSA blended PSf membranes.

layer pores) due to the higher polymer concentration of the casting solution [37,38] and also the homogeneously miscible additive inside bulk PSf structure made the pores smaller.

Moreover repelling BSA molecules (hydrophobic) from the membrane surface due to the more hydrophilic membrane structures of the P1 and P2 may be the another factor on increase the rejection ratio [39]. Also, the decrease on the rejection performance despite the thickness of the P3 membrane top layer increased can be explained by the disrupting the regular pore structure due to the comparatively high additive content in solution composition. Probably the interactions between the PANI backbone and additive that given in Fig. 1, may caused disrupting effect inter-chain structure of PANI backbone for comparatively high ratios (0.5 %). On the other hand, the rejection results showed that all the prepared PANI-PAMPSA blended PSf membranes had higher than 90 % rejection so the results indicated that all the membranes had MWCOs lower than 66 kDa. Therefore the PANI-PAMPSA blended PSf membranes can be classified as UF membrane.

4. Conclusion

In this work, novel high permeable PANI-PAMPSA blended PSf ultrafiltration membranes prepared. IR spectra of the membranes confirmed the interactions between PSf and PANI-PAMPSA, also significant increases in the OH stretching and bending frequencies attributed to higher water adsorption capacities and thus the more hydrophilic structure of the blend membranes that also confirmed contact angle results. SEM images showed that the main differences occurred on the cross-sectional structures. Finger-like pore lengths increased with the addition of PANI-PAMPSA, with the %0.25 additive amount, almost all throughout the membrane cross-sectional structure turned into finger-like pores that attributed to instantaneous demixing. Then, 0.5 % additive amount caused to formation of the shorter finger-like pores and macro void formations observed beneath the short finger-like pores. On the other hand the skin layer thicknesses of the membrane decreased with the 0.1 and 0.25 % additive amount. Dynamic contact angle measurements revealed the PANI-PAMPSA additive increased the surface hydrophilicity of the PSf membranes. Ultrafiltration tests demonstrated that PANI-PAMPSA additive had permeance enhancing effect of the PSf membrane, especially for low additive amounts (0.1 and 0.25 %), however, 0.5 % additive ratio caused a small decrease, was still higher than pristine PSf membrane permeance. The increase in permeability of the P1 and P2 membrane attributed to longer finger-like pore structure on the cross-section of the membranes and the higher hydrophilic character of the membrane owing to the hydrophilic additive. The permeance decline on P3 membrane most probably caused by the thicker dense skin layer of the membrane and also pore-blocking phenomena due to the relatively higher ratio additive. The rejection ratios of the prepared membranes increased from 90.1 (P0) to 95.8 and 98.4 % for P1 and P2 membranes then decreased to 95.2 % for P3 membrane. The increase on rejection of the P1 and P2 attributed to the formation of membrane structure with smaller pores (especially skin layer pores) due to the higher polymer concentration of the casting solution and also the homogeneously miscibled additive inside bulk PSf structure made pores smaller. Therefore the study showed that the PANI-PAMPSA is an effective additive on PSf membrane hydrophilicity and performance. All the blend membranes had higher than 90 % rejection performances for BSA and the results indicated that all the membranes had MWCOs lower than 66 kDa so the PANI-PAMPSA blended PSf membranes can be classified as UF membrane.

CRediT authorship contribution statement

Adem Sarihan: Conceptualization, Methodology, Software, Data curation, Writing - original draft, Visualization, Investigation, Supervision, Validation, Writing - review & editing.

Acknowledgements

We thank to “Scientific and Technological Research Council of Turkey (TUBITAK) BIDEB-2219 International Postdoctoral Research Fellowship Programme” for financial support. And also we thank to the technician team of Bath University, Chemical Engineering, Physics and Chemistry departments, for instrumentation.

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