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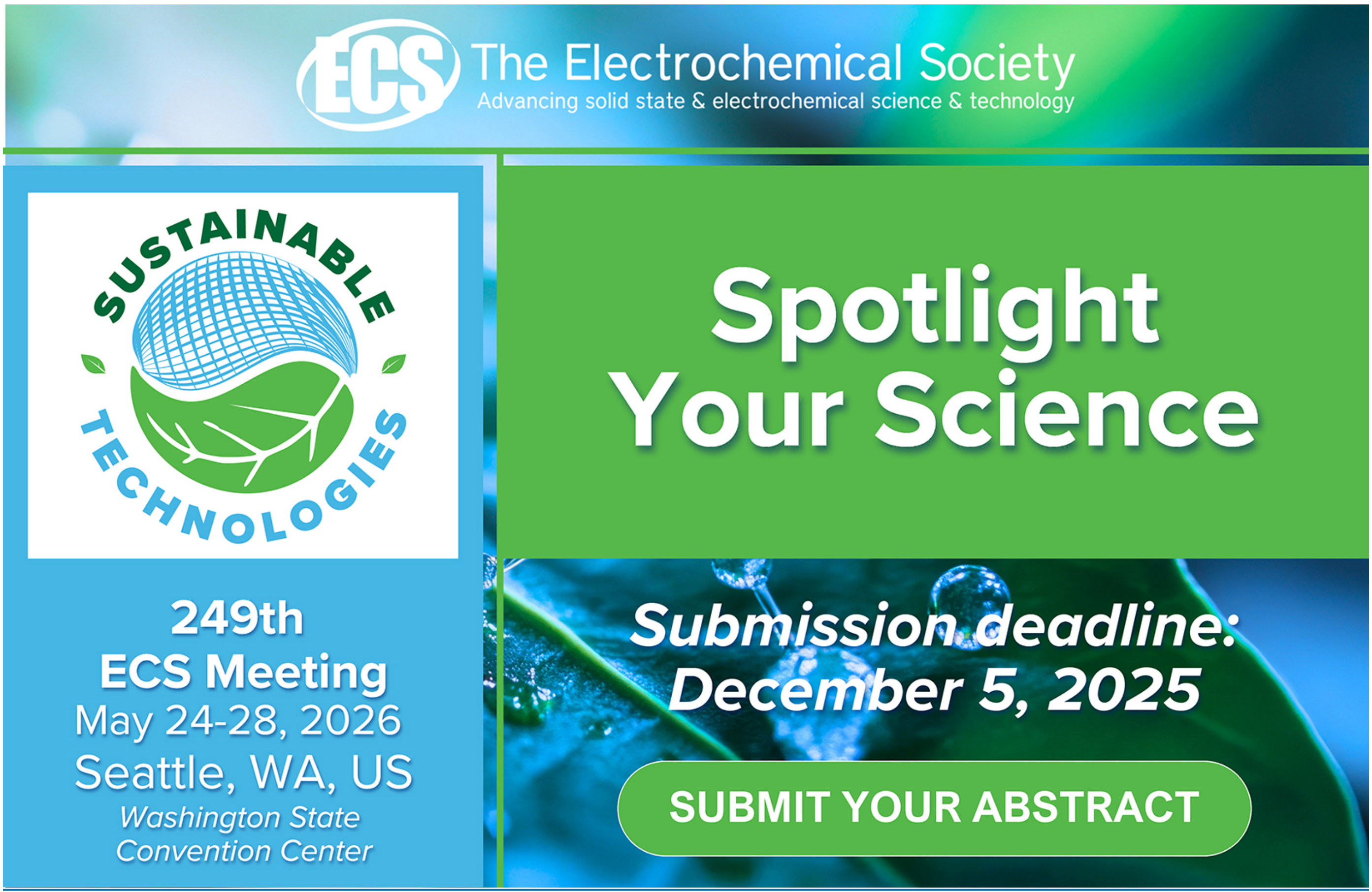

Physicochemical properties of PSF/SiO_2-NH_2 composite membranes incorporated by polymeric additives

T Ellis, C Sricheewin and C Yuenyao*

Department of Physics, Faculty of Science and Technology, Phetchabun Rajabhat university, 83 M. 11 Saraburi-Lom sak Road, Sadiang Sub-district, Muang Phetchabun 67000, Thailand

*Corresponding author's e–mail address: chalad.yue@pcru.ac.th

Abstract. This research aims to study the effects of PVP and PEG on the morphological structure, hydrophilicity and mechanical properties of PSF/SiO_2-NH_2 composite membranes. PSF/SiO_2-NH_2 membranes with and without the addition of PVP and PEG are fabricated by phase inversion process and used to study the physical and chemical properties as mentioned. Change of the structure and wettability properties of prepared membrane samples are determined by different scientific techniques. Hydrophilic properties are evaluated through the measurement of water contact angle (WCA) while morphology and pore size, as well as the creation of pore on the top surface of membrane samples are analyzed through the SEM micrographs. Additionally, alteration of hydrophilic functional groups on the top skin surface of the membrane is measured by FTIR technique. Experimental results illustrated the effects of PVP and PEG on the morphological and porous structures of PSF/SiO_2-NH_2 composite membranes. Internal porous structure have clearly changed from the sponge-like structure with macrovoids to finger-like structure. Macrovoids disappeared with the addition of PVP and PEG into the matrix of PSF/SiO_2-NH_2 composite membranes. Experimental results also showed that nanopore created on the top surface of PSF/SiO_2-NH_2 membranes incorporated with PVP and PEG. Importantly, PVP and PEG show the influence to the hydrophilic properties of PSF/SiO_2-NH_2 membranes. The effect on the hydrophilic properties as mentioned is confirmed by the increasing of hydrophilic group on the membrane surfaces and decreasing of WCA. DMTA results showed that PVP and PEG affect the glass transition temperature and mechanical properties of PSF/SiO_2-NH_2 composite membranes.

1. Introduction

Polymers, ceramics, metal, carbon and glass can be used as a thin membrane. Nowadays, most of commercial membranes are fabricated from polymeric materials. A very popular polymer is plastic, synthetic polymer and nowadays, polymers are developed to have good flexibility [1]. In the past decade, membrane separation technology has been widely used in the food industry, medicine, biotechnology because it is a low energy consumption technology, no phase change during separation. Phase inversion process or polymer phase transition is widely used to prepare porous polymer membranes. Polymeric additives with good membrane properties are considered to be quite effective additives. An important effect of inherent hydrophilic additives is to increase the water (hydrophilic) solubility on the membrane surface, stop building a large cavity (macrovoid) and optimization of inter- lockable porosity (interconnectivity). Polyethylene glycol (PEG) is a synthetic chemical in a group of organic substance with high hydrophilic properties. Therefore, PEG is used as an additive to enhance the hydrophilicity of different types of membranes. Polyvinyl pyrrolidone (PVP) can be dissolved in water and other polar solvents. PVP also dissolves in a various types of alcohols such as methanol, isopropanol, ethanol and other solvents. The incorporation of PVP in the dope solution affects the wetting properties of resulted films and make the film easier to form. Silicon dioxide (SiO_2) is used as a catalyst component, used as a strength enhancer and density in rubber, plastics and polymer products, etc. The amine-added SiO_2 is intended to make SiO_2 more hydrophilic and more porous.

This research project attempted to study the effects of PVP and PEG on the morphological structure, hydrophilicity and mechanical properties of PSF/SiO_2-NH_2 composite membranes. Pure PSF and composite of PSF/SiO_2-NH_2 membranes with and without the addition of PVP and PEG are fabricated by phase inversion method. Alteration of characteristics of synthesized membrane samples are evaluated by different analytical techniques. According to a review of the study papers, there have been no studies on the characteristics of PSF/SiO_2-NH_2 composite membranes incorporated with PVP and PEG.

2. Materials and methods

N-methyl-2-pyrrolidone (NMP, C_2H_9NO, 99.0% purity, M_W: 99.13 g/mol), polyvinyl pyrrolidone (PVP360, average molecular weight of 360,000) and 3-aminopropyl triethoxysilane (APTES) were supplied by Sigma-Aldrich (USA). Polyethylene glycol (PEG1000), tetraethylorthosilicate (TEOS, M_W: 208.33 g/mol) and cetyltrimethylammonium bromide (CTAB, >98.0% purity, M_W: 364.45 g/mol) were supplied by Merck, Thailand. Pellet of PSF materials (Udel P-3500 LCD MB) were supported by Solvay, China. Ethanol (EtOH, AR. Grade) and cyclohexane (CHX, C_6H_{12}, M_W: 84.16 g/mol) was supplied by RCI-LabScan. L-Arginine (LAG, M_W: 174.20 g/mol) was supplied by Loba Chemie, India. SiO_2-NH_2 used in this work was synthesized by co-condensation method under water-CHK biphasic condition [2]. All of chemicals were used as received.

All of membranes in this work were prepared by a dry-wet phase inversion method [3]. Five types of membrane including PSF, PSF/PEG, PSF/PVP, PSF/PEG/SiO_2-NH_2, and PSF/PVP/SiO_2-NH_2, were fabricated. For pure PSF membrane preparation, 18 g pellet of PSF materials was dissolved in 82 g NMP at 60 °C for 18 h. PSF incorporated with PEG and PVP membranes were prepared with the same process by controlling of PEG and PVP contents at 1.0, 2.5 and 5.0 wt% of PSF content. To fabricate PSF composite membranes, polymeric additive were dissolved in the NMP and pellet of PSF was added to the system after the additives completely dissolve in the solvent. SiO_2-NH_2 in the membrane system was controlled at 1.0 wt% of PSF content. Content of PSF in all membrane was controlled at 18 wt% while the content of NMP depend on the additive loading. To study the alteration of morphological structure and mechanical properties of PSF composite membranes, SEM and DMTA were employed, respectively. Hydrophilicity and variation of functional groups on the top skin membrane surface were evaluated through the measurement of water contact angle (WCA) and FTIR spectrum.

3. Results and discussion

Table 1. Water contact angles (WCAs) of top skin membrane surfaces.

Membrane names	Water contact angle (WCA, °)
Pure PSF	87.35
PSF/SiO_2-NH_2 (1.0 wt%)	77.72
PSF/SiO_2-NH_2/PEG(1.0 wt%)	79.38
PSF/SiO_2-NH_2/PEG(2.5 wt%)	74.14
PSF/SiO_2-NH_2/PEG(5.0 wt%)	71.08
PSF/SiO_2-NH_2/PVP(1.0 wt%)	79.66
PSF/SiO_2-NH_2/PVP(2.5 wt%)	77.18
PSF/SiO_2-NH_2/PVP(5.0 wt%)	76.42

Measurement of WCA results, as shown in table 1, and increase of hydrophilic functional groups (hydroxyl; –OH, and carbonyl; –CO, groups) as shown in figure 1, on the top surface of PSF composite membranes confirmed the increase of hydrophilicity. These results indicated that WCA of membrane surfaces decrease with increase loading of polymeric additives.

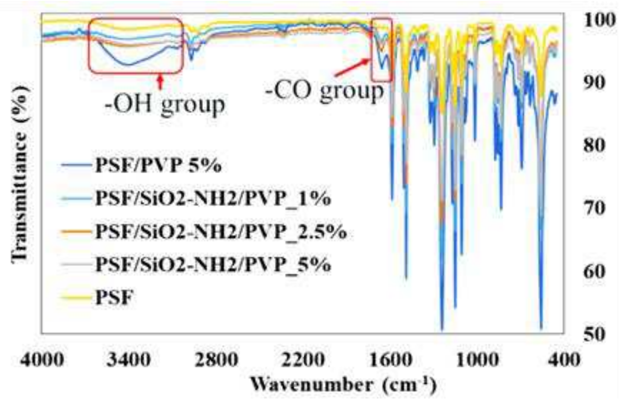

Figure 1. FTIR spectrums show the change of functional groups on top surface of pure and composite PSF membranes.

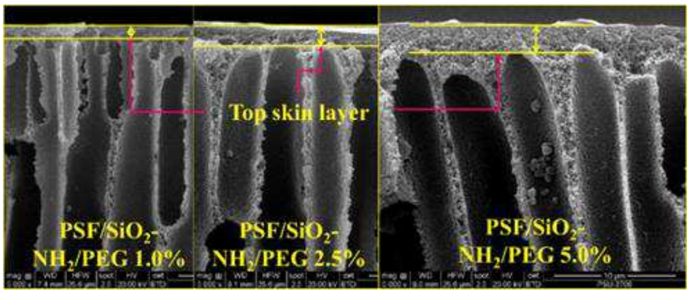

Figure 2. Comparison of the top skin layer thickness of PSF/SiO_2-NH_2 composite membranes incorporated with PEG at different concentrations.

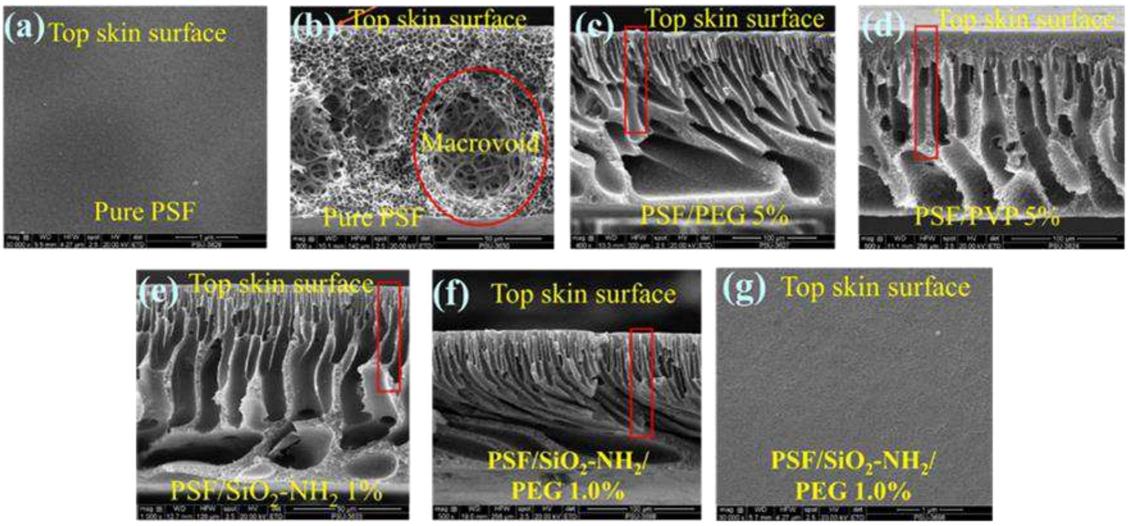

Figure 3. Morphology of top skin surface of (a) pure PSF and (g) PSF/SiO_2-NH_2/PEG 1.0 wt% and cross section view of (b) pure PSF, (c) PSF/PEG 5.0 wt%, (d) PSF/PVP 5.0 wt%, (e) PSF/SiO_2-NH_2 1.0 wt% and (f) PSF/SiO_2-NH_2/PEG 1.0 wt% membranes.

SEM micrographs as shown in figure 2 shows the increase of top skin layer thickness of PSF/SiO_2-NH_2 composite membranes when PEG loading increase from 1.0 to 5.0 wt%. In addition to the increase of top skin layer thickness, SEM micrographs (as shown in figure 3) also show the alteration of morphological structure of PSF composite membranes. It was found that macrovoids and sponge-like structure of pure PSF membrane as shown in figure 3(b) clearly replaced with finger-like structure and a small portion of sponge-like structure in between finger-like structures when PEG, PVP, SiO_2-NH_2 and SiO_2-NH_2/PEG are incorporated in the matrix of PSF membrane. In this study, dense top skin surface was created on the pure PSF, PSF/PEG, PSF/PVP and PSF/SiO_2-NH_2 membranes. As can be seen from figure 3(a) and 3(g), the dense top skin surface of pure PSF was replaced by non-dense skin surface with some pores when SiO_2-NH_2/PEG 1.0 wt% incorporated in the matrix of membrane. The results from this study showed that the effects of PEG and PVP on the morphology and hydrophilicity of PSF membranes resemble with reported in literatures [4–6].

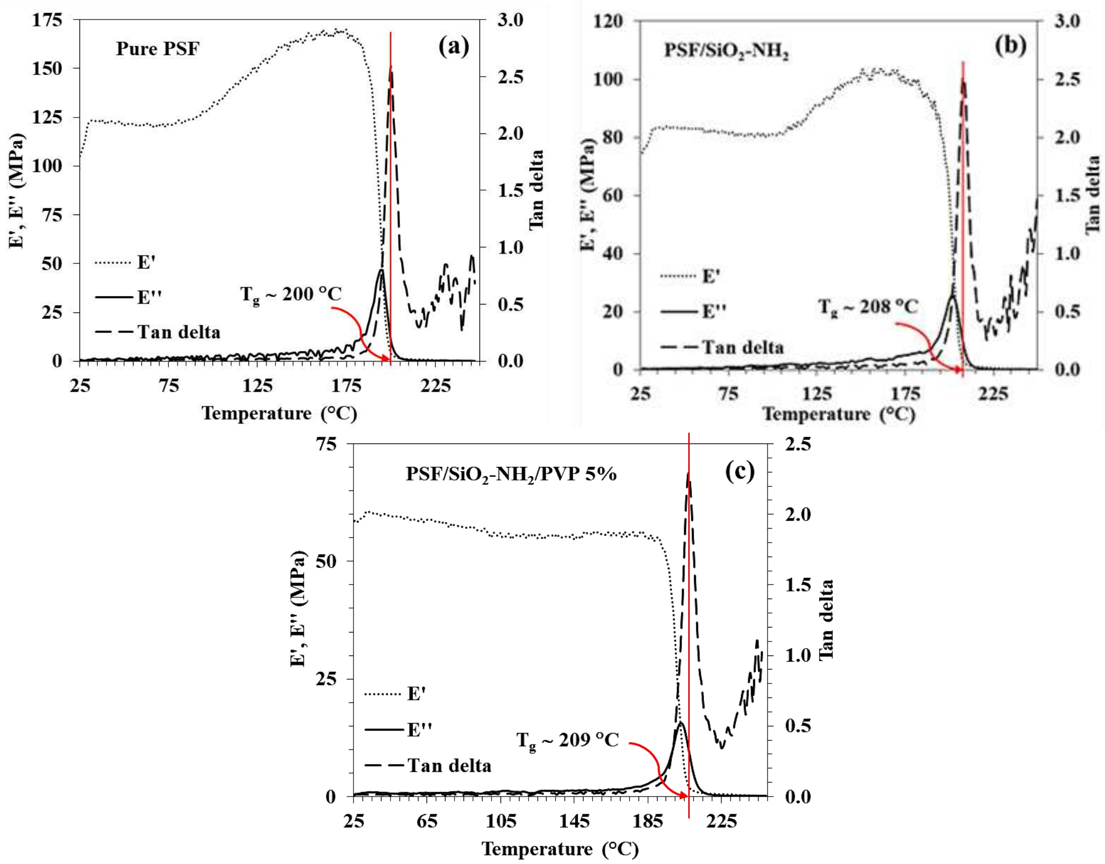

Figure 4. DMTA results show the alteration of viscoelastic property and glass transition temperature (T_g) of (a) pure PSF, (b) PSF/SiO_2-NH_2 and (c) PSF/SiO_2-NH_2/PVP composite membranes.

DMTA results as shown in figure 4 confirmed the good interaction of polymeric additives and matrix of PSF and also showed the alteration of viscoelastic property and glass transition temperature (T_g) of composite membranes. Incorporation of PEG and PVP in the matrix of PSF lead to decrease of T_g. Addition of SiO_2-NH_2 and SiO_2-NH_2/PVP can increase T_g of PSF membrane from about 200 ºC to 208 ºC and 209 ºC, respectively. In contrast, addition of SiO_2-NH_2/PEG lead to decrease of T_g of PSF membranes. The PSF composite membrane with thin and dense top skin layer, high hydrophilicity, high porous structure, regular finger-like structure and high mechanical strength is desired in this research.

4. Conclusion

In summary, morphological structure and mechanical strength of PSF membranes can be adjusted by incorporation of inorganic and organic additives into the matrix of membranes. Hydrophilic properties can be controlled by the loading of polymeric (PVP and PEG) additives. Inorganic nanoporous and polymeric additives clearly affect the internal structure of PSF membranes. Macrovoids in pure PSF membrane were completely replaced by a finger-like structure in PSF composite membranes. Thickness of top skin layer can be controlled by variation of polymeric additives loading.

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