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Preparation and application of polyvinylidene fluoride/polyphenylene sulfone blending membrane

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ABSTRACT

In this study, the effects of PVDF/PPSU blend ratio, polymer concentration and additives (LiCl, $MgCl_2$, PEG-2000) concentration on PVDF/PPSU blending membrane performances were studied by orthogonal experiments, optimal blending membranes were obtained by orthogonal polar difference analysis and were investigated by treating esterification wastewater (pre-processed) through the submerged MBR. The results showed that with PVDF/PPSU blend ratio changed from 9:1 to 5:5, water flux increased but rejection and contact angle decreased, with the polymer concentration changed from 11% to 19%, water flux and contact angle decreased but rejection went up, with the additive concentration increased, water flux and rejection slightly change but contact angle decreased obviously. The best water flux, rejection and contact angle were 3,300 L m^{-2} h^{-1}, 60% and 67°, respectively. The membrane was adopted to treat esterification wastewater (pre-processed) by MBR, which had the best performance with 1 wt.% PEG-2000 concentration added and 19 wt.% polymer concentration (PVDF:PPSU was 5:5), steady treatment efficiency was obtained that the COD_{cr} removal rate was more than 75% and the highest COD_{cr} removal rate was 84.3%.

Keywords: PVDF/PPSU membrane; Membrane bioreactor; Esterification wastewater

1. Introduction

In recent years, with the rapid progress of chemical industry and enhancement of environmental consciousness, more and more attention has been placed at esterification wastewater treatment. Because of low pH (<4), high COD_{cr} (>18,000 mg L^{-1}) and complex components (terephthalic acid, polyester particles and molecules colloids), esterification wastewater was difficult to treat and posed severe threatening to the environment [1–3]. At present, more and more wastewater treatment technologies have been applied in the fields of esterification water, such as chemical treatment, biological treatment and membrane method [4–6]. Compared with the chemical and biological treatment method, the membrane method has many advantages such as anti-impulsion load, stable treating effect, simple running management; especially, the membrane bioreactor (MBR, which combined of the conventional activated sludge process and ultrafiltration membrane) is most widely adopted to treat esterification wastewater due to its excellent solid–liquid separation and operating efficiency, less site area, low sludge production and easy to realize automatic operation [7]. For MBR module, membrane is the key element of treatment process; however, membrane bioreactors are still greatly restricted by membrane fouling, which effect the process performance such as energy consumption, membrane life and water production capacity. Therefore, a suitable membrane is essential required to

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enhance MBR treatment efficiency which have superior permeability, hydrophilicity and chemical resistance [8].

Polyvinylidene fluoride (PVDF) has received great attention in the ultrafiltration membrane preparation for its outstanding properties such as excellent thermal stability, chemical resistance to aggressive reagents (organic solvents, acid and bases), highly organic selectivity, as well as good mechanical strength and membrane forming properties [9–11]. Generally speaking, PVDF can be dissolved in many common organic solvents such as N-dimethylformamide (DMF), N-methylpyrrolidone (NMP) N,N-dimethylacetamide (DMAc) and dimethyl sulfoxide (DMSO). Compared with amorphous polymer, PVDF generally exhibits more complicated phase separation behaviour as a semi-crystalline polymer [13–15]. However, due to its hydrophobic nature and low surface energy, some impurities would adhere to the surface of PVDF membrane which would block pores and results in severe membrane fouling, flux reduces and operation cost increases [16,17]. Some measures which have been carried out to modify PVDF membrane performance include physical blending, chemical grafting, and surface modifications [18]. However, the most used method is physical blending because of the materials' convenient operations, mild conditions and good performances. Generally speaking, polymer, macromolecules, non-solvents and inorganic can be blended as additives, which can suppress or excite the formation of macrovoids, influence pore interconnectivity and hydrophilicity, modify membrane performance [19,20].

Polyphenylene sulfone (PPSU) is known as one of polysulfone materials, which has an excellent chemical resistance, outstanding mechanical strength, thermal resistance and non-toxic. In its molecules, there is no methyl structure influencing the steric hindrance; the formation in which benzene rings are directly connected maintains material rigidity, and ether bond is connected with molecules on both sides, which enhances the flexibility and mobility of PPSU molecules [21,22].

In this study, PVDF/PPSU blending membranes were prepared by phase inversion method. The effects of PVDF/PPSU blend ratio, polymer concentration and additives (LiCl, $MgCl_2$, PEG-2000) concentration on membrane performances were studied. Water flux and rejection were investigated, contact angle for a liquid drop on the membrane surface were measured to express the surface wettability of membranes. SEM images were used to compare inner structures and pore spread of differences of optimal blending membranes. Meanwhile, Optimal blending membrane was used in the submerged MBR module to treat esterification wastewater (pre-processed), COD_{cr} removal rate was observed as the evaluation index.

2. Materials and methods

2.1. Materials

PVDF (relative molecular mass of 444,000) was used as the main membrane material; PPSU (chemical pure, Basf) was also used as another membrane material. DMAc (N,N-Dimethylacetamide, relative molecular mass of 87.12, analytical reagent, Beijing chemical works) was used as the polymer solvent. LiCl (Lithium chloride, anhydrous, relative molecular mass of 42.39, Jinke fine chemical industry research institute), MgCl2 (magnesium chloride, anhydrous, 99%) and PEG-2000 (polyethylene glycol-2000) were adopted in the preparation of the PVDF/PPSU membranes as the non-solvent additives. All the additions were dried at 50°C at least 24 h to eliminate the absorbed water molecules before used, blood serum albumin (BSA; Shanghai Bio Life Science & Technology Co., 98%).

2.2. PVDF/PPSU blending membrane preparation

In this study, PVDF/PPSU blending membranes were prepared by phase inversion method. PVDF, PPSU and additive were dissolved in DMAc; the mixture was dissolved at 55°C until to obtain homogeneous polymer solution. The solutions were cast onto clean glass plate at room temperature, and the thickness of the blending membrane was controlled by using 0.2 mm casting knife. Then, the liquid membranes and the glass plate were moved toward the nonsolvent bath (distilled water) for immersion precipitation at room temperature immediately. After the membrane peeled off from the glass plate, which would been carried out after further immersed in distilled water for 24 h.

2.3. Experimental orthogonal array

In this study, $L_{25}(5^6)$ orthogonal array was adopted to conduct experiments, three influence factors: PVDF/PPSU blend ratio (A), polymer concentration (B), additives (LiCl, $MgCl_2$, PEG-2000) concentration (C) were selected, and each factor had five levels. Based on the orthogonal test system, the influence of PVDF/PPSU blend ratio, polymer concentration and additives (LiCl, $MgCl_2$, PEG-2000) on water flux, rejection and contact angle were tested, the optimal combination of influence factors was determined. The experimental orthogonal array was shown in Table 1.

2.4. Characterizations of membrane

The PVDF/PPSU blending membranes permeation performance, rejection performance and hydrophilicity were conducted using water flux, rejection and contact angle, respectively. Water flux was observed with a membrane piece surface area of 0.0026 cm^2. When the flux was steady,

Table 1
Experimental orthogonal array

Level	Factor				
	A (PVDF/PPSU blend ratio)	B (Polymer concentration) wt.%	C (Additive) wt.%		
			LiCl	$MgCl_2$	PEG-2000
1	9:1	11	1	1	1
2	8:2	13	1.3	1.3	4
3	7:3	15	1.6	1.6	7
4	6:4	17	1.9	1.9	10
5	5:5	19	2.2	2.2	13

membrane characterization of the water flux for PVDF/PPSU blending membranes was measured at 0.1 MPa, 25°C, then calculated by the following equation: $F = V / (AT)$, where V is the volume of the permeate in liters, A is the membrane surface area (m), and T is the permeation time (h), and F is the water flux (L m^{-2} h^{-1}).

Rejection was characterized with 0.5 g L^{-1} BSA aqueous solution after the membrane was previously filtered with pure water at 0.1 MPa, 25°C. The rejection of protein was obtained by: $R = (1 - C_p / C_j) \times 100$ (%), where C_p is the BSA concentration of the permeate and C_j is the concentration of the feed.

The hydrophilicity of PVDF/PPSU blending membrane was examined using contact angle measurement (JC2000D). A water droplet was placed on a flat homogeneous membrane surface and the contact angle between the water and membrane was measured until no change was observed. To minimize the measurement error, a total of six replicates were taken and averaged. The cross-sectional and inner surface morphology of PVDF/PPSU blending membranes was observed and performed using a scanning electron microscope (ESEM, QUANTA2000, FEI).

2.5. MBR experiments

Esterification wastewater are treated by MBR module which used the optimal PVDF/PPSU blending membrane, and the esterification wastewater was pre-processed (pretreatment – anaerobic/aerobic treatment – advanced oxidation) to obtain a suitable COD_{cr} for MBR reactor. Installation drawing of MBR was showed (Fig. 1). COD_{cr} as the evaluating indicator to test the treatment efficiency of optimal membranes in MBR module. Esterification wastewater from a factory in Huanghua, Hebei province, was used in this study. The pH value, COD_{cr} and turbidity of the esterification wastewater used in this study were 2~3, 70,000~90,000 mg L^{-1} and 0.41~1.7 NTU, respectively; before wastewater flow into MBR, some treating process (pretreatment – anaerobic/aerobic treatment – advanced oxidation) were adopted in order to avoid overload of MBR module happen.

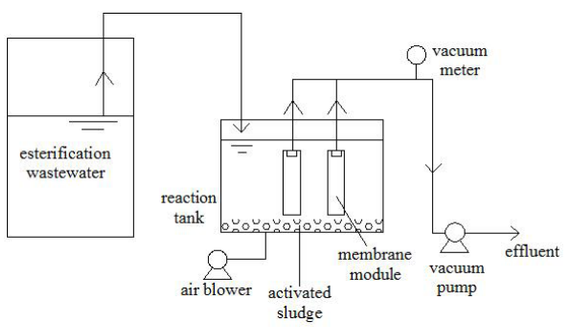


Fig. 1. Schematic drawing of MBR.

3. Results and discussion

3.1. Optimal combination of membranes

Orthogonal polar difference analysis result was shown in Table 2. It can be seen that when additive were LiCl, $MgCl_2$ and PEG-2000, polymer concentration is the main factor affecting water flux, influence of blend ratio was higher than additive concentration, the optimal membrane solution compositions (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of the largest water flux were 5:5/11% polymer/2.2% LiCl (wt.%); 7:3/11% polymer/1.6% $MgCl_2$ and 7:3/11% polymer/7% PEG-2000 (wt.%), respectively.

When LiCl, PEG-2000 were added, respectively, rejection was mainly influenced by polymer concentration, additive concentration took the second place, the optimal membrane solution composition (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of the largest rejection was 7:3/19% polymer/1.3% LiCl (wt.%) and 8:2/19% polymer/1.0% PEG-2000 (wt.%). In terms of $MgCl_2$, polymer concentration was the main factor affecting rejection, the influence of blend ratio was higher than additive concentration and the optimal composition (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of the largest rejection was 9:1/17% polymer/1.9% $MgCl_2$ (wt.%).

When additive was LiCl, polymer concentration was the main factor of contact angle, the influence of LiCl concentration was higher than blend ratio, the optimal membrane solution composition (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of contact angle was 7:3/19% polymer/1.3% LiCl (wt.%). When additive was $MgCl_2$, polymer concentration and blend ratio were primary and secondary factor, respectively, the optimal membrane solution composition (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of contact angle was 8:2/19% polymer/1.0% $MgCl_2$ (wt.%). As for PEG-2000, its concentration influenced contact angle mainly; the optimal membrane solution composition (PVDF/PPSU blend ratio, polymer concentration and additives concentration) of contact angle was 9:1/11% polymer/13% PEG-2000 (wt.%).

Via orthogonal polar difference analysis, optimal level and combination were obtained. The optimal combina-

Table 2
Orthogonal polar difference analysis

Index	Polar difference	LiCl			$MgCl_2$			PEG-2000		
		A	B	C	A	B	C	A	B	C
Water flux (L m^{-2} h^{-1})	R_1	702	1,865	514	1,999	2,557	617	1,391	2,555	887
Rejection (%)	R_2	12.7	21.5	15.6	14	34	6	4.40	32.2	5.70
Contact angle (°)	R_3	4.33	15.4	6.23	6.24	13.9	2.86	7.68	8.7	10.5

tions (PVDF/PPSU blend ratio, polymer concentration and additives concentration) were 5:5/17% polymer/1.6% LiCl (wt.%), 7:3/19% polymer/1.0% $MgCl_2$ (wt.%) and 5:5/19% polymer/1% PEG-2000 (wt.%).

3.2. Morphologies of optimal PVDF/PPSU blending membrane

The morphology of the membranes was observed by SEM to represent the cross-sectional at a certain magnification. In Fig. 2 (a), neat PVDF membrane (17 wt.%) has finger-like voids and small aperture. The structure of cross-section was shown in Fig. 2 (b), which was prepared without any additive (7:3/17% polymer wt.%), exhibited an asymmetric structure consisting of sponge-like substructure and thicker top layer of the membranes because when high entanglement degree between PVDF and PPSU polymer chains occurred, a dense cortex was formed during phase separation and porosity decreased. The morphologies of optimal membrane (5:5/17% polymer/1.6% LiCl wt.%) was shown in Fig. 2 (c), loose net-like and banded structure were observed, the presence of LiCl favored the formation of larger and longer macrovoids, the dope's thermodynamic instability increased in the reaction with water, which contributed to a rapid phase separation and resulted in macrovoid formation, and water flux increased remarkably. The membrane (7:3/19% polymer/1.0% $MgCl_2$ wt.%) was clearly observed with finger-like pores in top skin layers, and macrovoids structure and banded structure in the sub layer (Fig. 2 (d)). The above observations were believed to be associated with the change of the thermodynamic and kinetic properties of the system because of the good affinity with water of $MgCl_2$. In Fig. 2 (e), the membrane (5:5/19% polymer/1% PEG-2000 wt.%) showed that thicker layers were formed in the membranes, and internal structure was larger and longer macrovoid, which is due to the hydrophilism and thermodynamics performance of PEG-2000.

3.3. Effect of PVDF/PPSU blend ratio on membrane performance

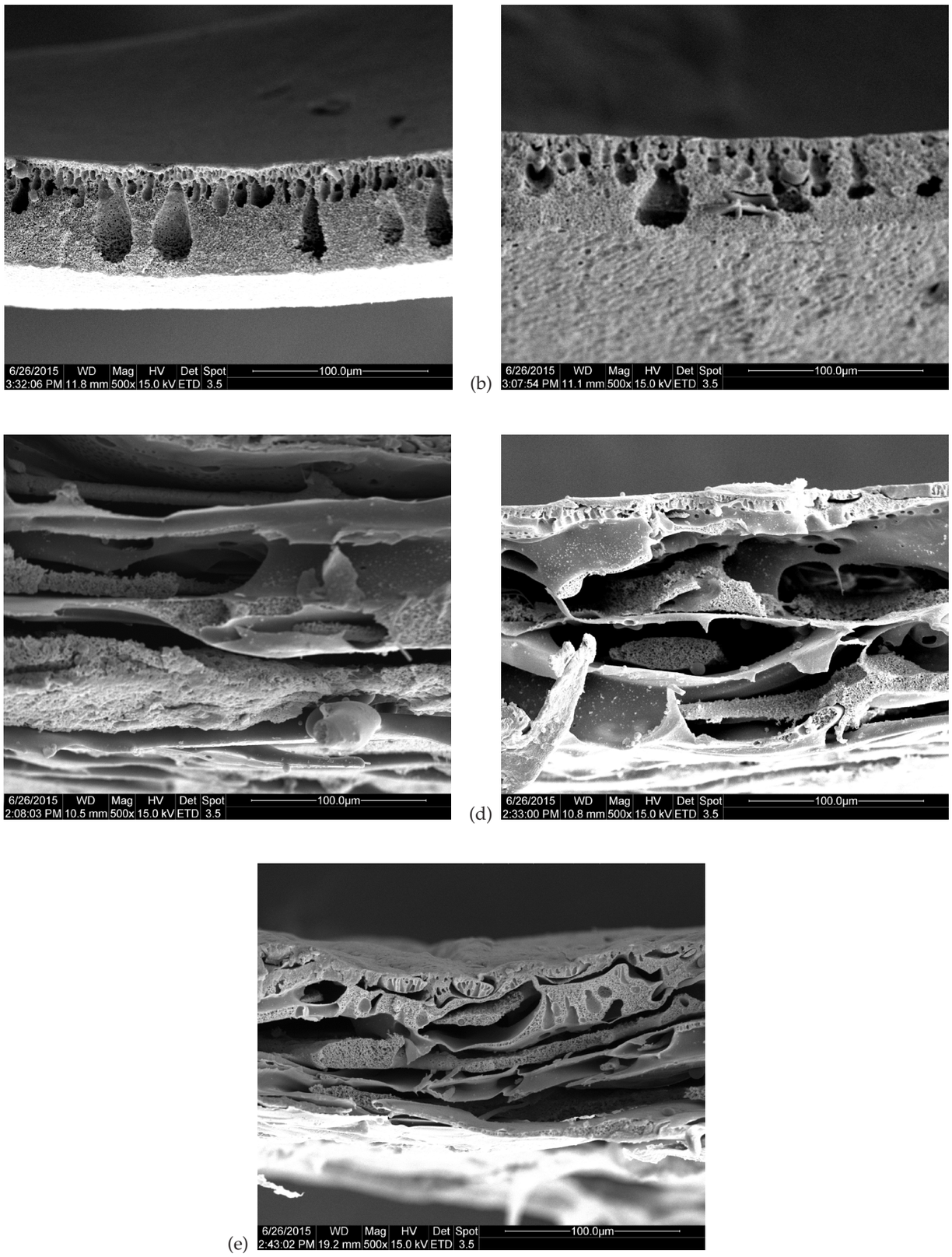
The influence of polymer blend ratio on membrane performance was studied (Figs. 3–5). When LiCl was added as additive, water flux was minimum compared with others, but rejection was maximum on the whole. When blend ratio changed from 9:1 to 7:3, water flux increased at first then decreased (from 715.22 to 1,056.28 L m^{-2} and then to 825.12 L m^{-2} h^{-1}), and maintained uptrend later (Fig. 4). This was because PVDF was easy to form large micellar aggregates in DMAc, viscosity and surface tension decreased with the decreasing proportion of PVDF in the mixture (Fig. 3), the interaction weakened between polymer and solvent, phase separation of polymer was promoted and finger-like pore formed so that the water flux increased. However, blend ratio had a little effect on contact angle (Fig. 5). When $MgCl_2$ was added, the change of blend ratio caused viscosity and surface tension drop off (Fig. 3); meanwhile, it enhanced water flux remarkably (from 669 to 2668 L m^{-2} h^{-1}), but rejection had a very small change (Fig. 4); the reason was that when PVDF and PPSU were mixed with different blend ratio with $MgCl_2$ added, numbers of membrane pores increased so that water flux increased, whereas the pore size was not changed and the ability of membrane to reject large molecules was not changed either. Contact angle had a tendency that decreased at first and then increased; the inflection point was 7:3. Water flux, rejection and contact angle were in turn-back type (i.e., the alteration trends is spiraling) fluctuation when additive was PEG-2000, water flux had a uptrend as a whole and the maximum water flux was 2,504 L m^{-2} h^{-1}, the reason might be that blend ratio changed, casting solution viscosity decreased (Fig. 3) led to the dissolution rate of polymer and additives accelerated, the membrane pores became more looser so that water flux increased. Contact angle had a slight increase with fluctuation, which indicated that the effect of polymer blend ratio on contact angle was small and unstable the reason for which might be individual differences appeared with the change of blend ratio. However, the membrane which added PEG-2000 had the smallest contact angle compared with others; it showed that the modification of the membrane hydrophilicity by PEG-2000 was the best.

3.4. Effect of polymer concentration on membrane performance

Polymer concentration was the main factor affecting water flux, rejection and even contact angle as discussed above. With polymer concentration increased from 11% to 19%, viscosity, surface tension and rejection increased obviously while water flux decreased (Figs. 6 and 7). When LiCl was added as additive, water flux decreased from 2,192 to 327 L m^{-2} h^{-1}, in terms of $MgCl_2$ and PEG-2000 system, water flux came down from 3,000 to 600 L m^{-2} h^{-1} approximately (Fig. 7), which is because the more polymer concentration, the higher the viscosity and entanglement degree between PVDF with PPSU polymer chains occurred, delayed diffusion rate of solvent and non-solvent, and phase separation process was inhibited, leading to dense cortical structure formed; the number of finger-like pores reduced, while more sponge-like pores were obtained, and pore size reduced, resulting in water flux decrease and rejection increase. Meanwhile, water flux of membrane which added PEG-2000 was the highest, and the reason might be that larger molecular weight of PEG-2000 was easier to form big and loose reticular pores, which made the water flux high. With the increase of the polymer concentration, contact angle of membrane with LiCl, $MgCl_2$ and PEG-2000 added were decreased from 87° to 69°, from 88° to 74° and from 78° to 69°, respectively (Fig. 8). The reason for this phenomenon is that with polymer concentration increased, phase separation rate slowed down; hydrophilic additives not only had more opportunities to combine with water but also had more entanglements with the polymer molecules, which led the additives to stay in membrane surface and inner; hydrophilicity of membrane was improved; therefore, contact angle decreased.

3.5. Effect of additive concentration on membrane performance

With the increase of LiCl concentration, water flux increased at first and then decreased; rejection decreased



(a) (b) (c) (d) (e)

Fig. 2. Morphologies of PVDF/PPSU blending membrane.

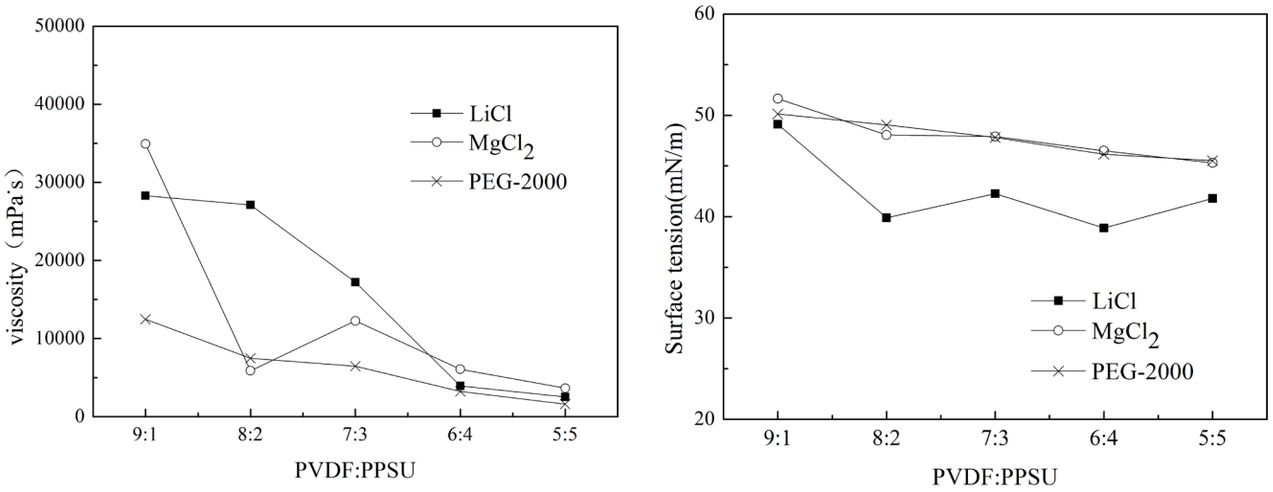


Fig. 3. Effects of PVDF/PPSU blend ratio on viscosity and surface tension with different additive.

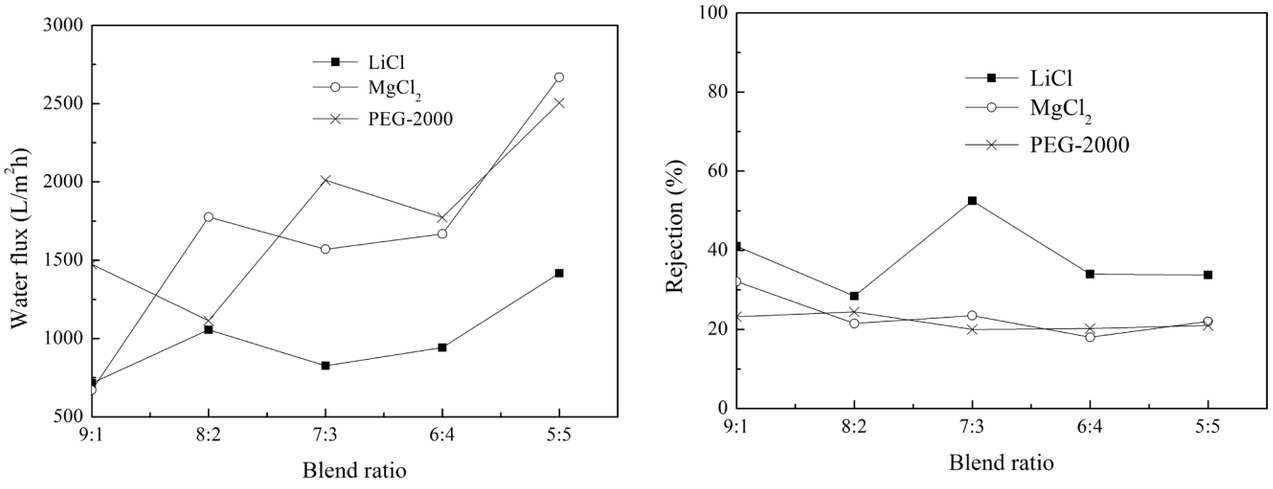


Fig. 4. Effects of PVDF/PPSU blend ratio on water flux and rejection with different additive.

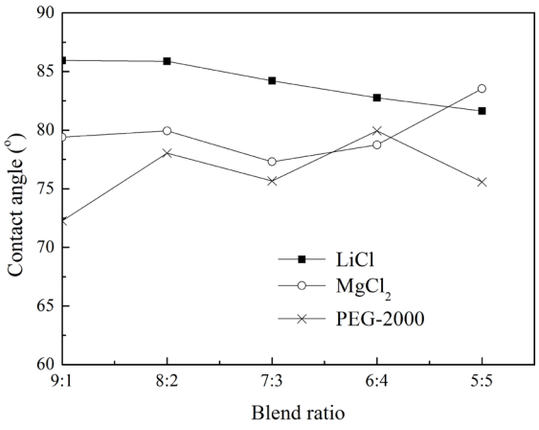


Fig. 5. Effects of PVDF/PPSU blend ratio on contact angle with different additive.

at first and then increased slowly. When LiCl concentration was 1 wt.%, smaller pores formed due to low additive concentration so that water flux was small but rejection and contact angle was high; when LiCl concentration was 1.3 wt.%, larger pores formed and pore size was the largest, and therefore water flux was the highest (1,223 L m^{-2} h^{-1}), and rejection was the lowest (31.4%). When LiCl concentration continued to increase to 1.3 wt.%, additive inhibited the formation of macropores and pores became smaller and fewer in size and number, leading to decrease in water flux, and it enhanced rejection and contact angle (Figs. 9–11). For $MgCl_2$ system, with $MgCl_2$ concentration changed from 1 wt.% to 2.2 wt.%, water flux increased and rejection maintained at 25%, when additive concentration was 1.6% and water flux and rejection were highest (Fig. 9). This was because $MgCl_2$ and polymer were compatible fully in solvent, dense pores with small size formed, and therefore water flux and rejection rate were great. When PEG-2000 concentration grew up to 7 wt.%, water flux increased to maximum remarkably

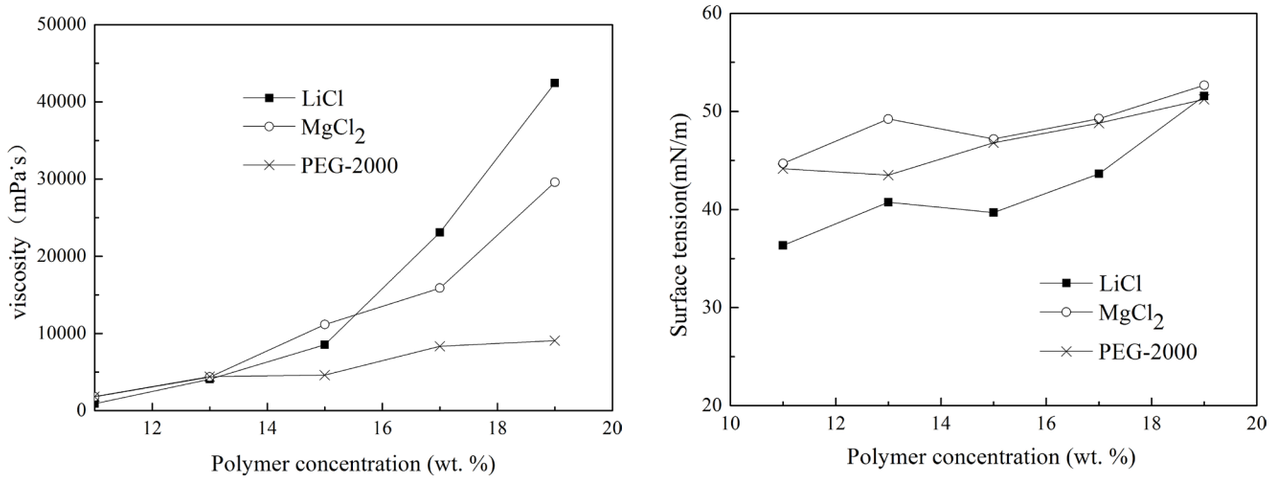


Fig. 6. Effects of polymer concentration on viscosity and surface tension with different additive.

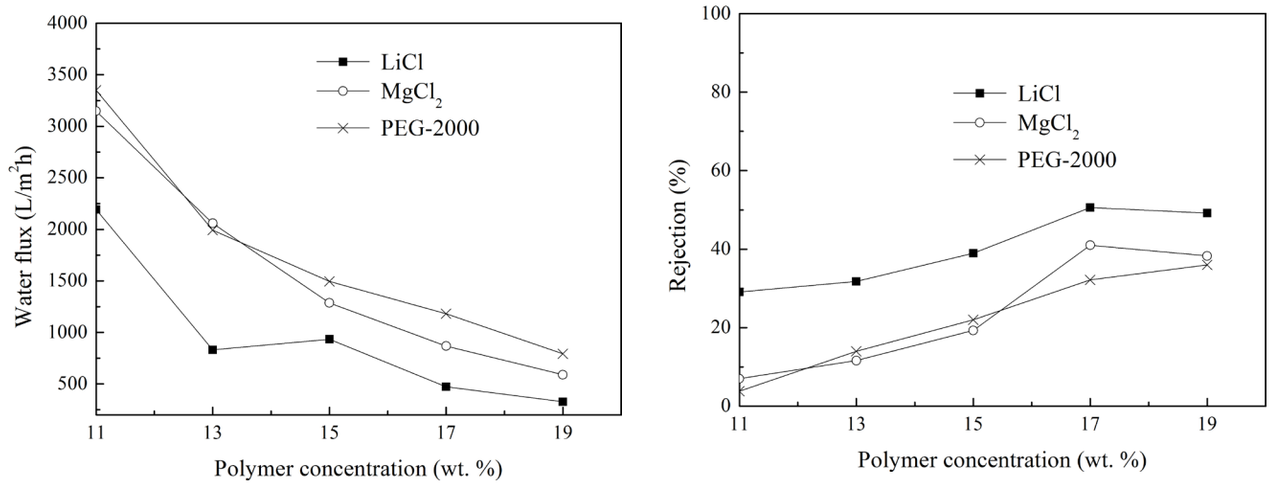


Fig. 7. Effects of polymer concentration on water flux and rejection with different additive.

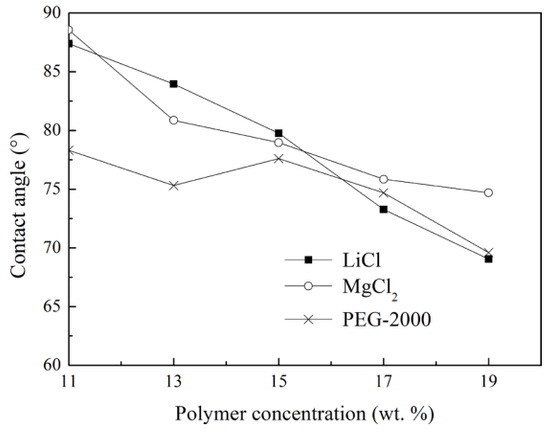
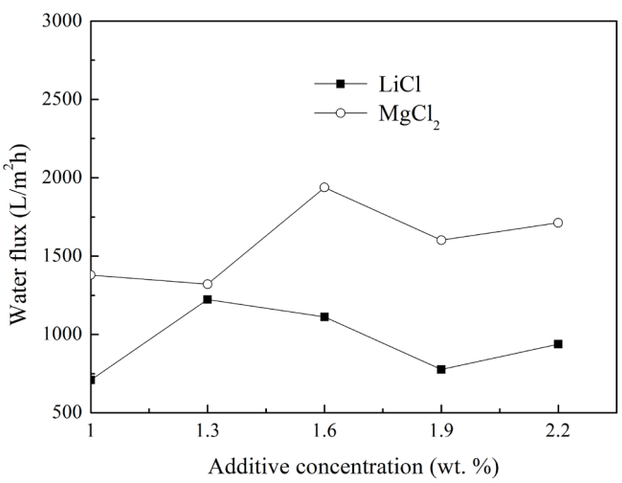


Fig. 8. Effects of polymer concentration on contact flux with different additive.

then decreased slightly with concentration increased from 7 to 13 wt.%, rejection decreased all the way (Fig. 10), which is due to the fact that PEG-2000 concentration increased to 7 wt.%, the most pores were induced by PEG-2000, macropore and loose network structure formed, water flux increased to maximum. When concentration increased to 13 wt.%, PEG-2000 might be dissolved incompletely and finger-like pores formed as dominant structure, resulting in a slight decrease in water flux. Meanwhile, in Fig. 12, contact angle which added PEG-2000 decreased remarkably (from 81° to 67°); it might be because with the increase of PEG-2000 concentration, the content of PEG-2000 in membrane pores also increased and hydrophilicity of the membrane improved. All the above indicated that additive concentration might promote or inhibit the formation of pores.

When additives were added in polymer solution, the solvent chemical's potential changes and the velocity of solvent exchanges diffusion speed, resulting in the formation of the membrane pore size and its distribution



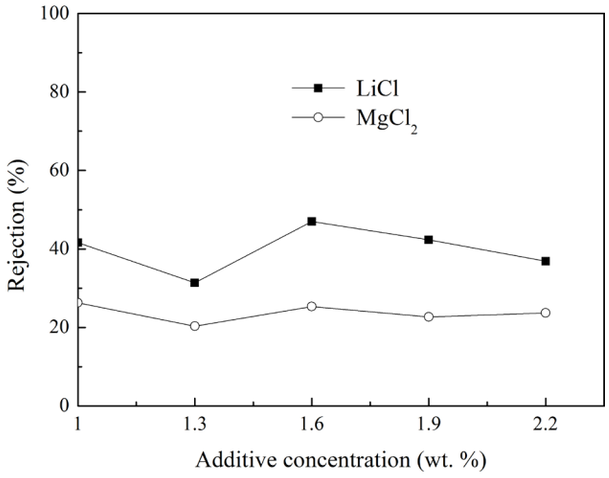


Fig. 9. Effects of additive concentration on water flux and rejection with different additive (LiCl and $MgCl_2$).

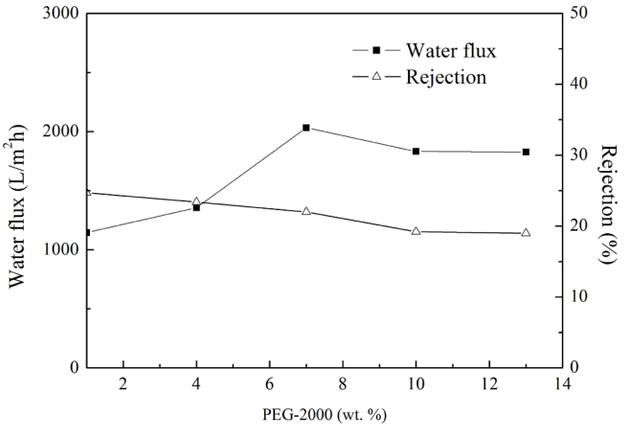


Fig. 10. Effects of additive concentration on water flux and rejection (PEG-2000).

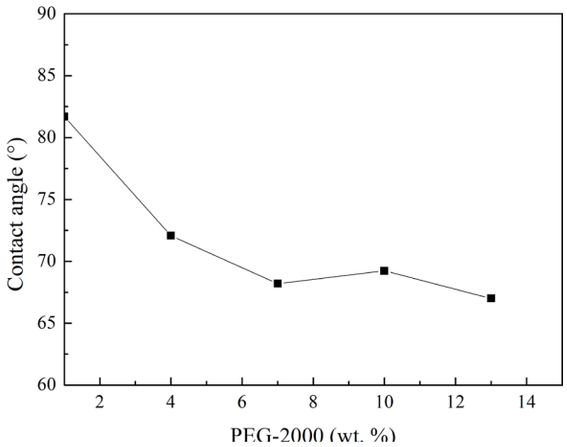


Fig. 12. Effects of additive concentration on contact angle.

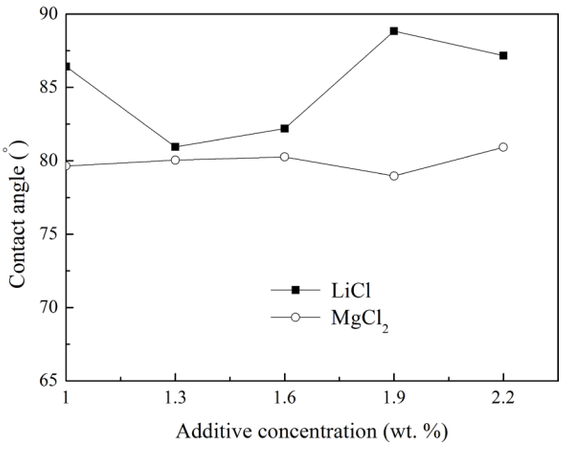


Fig. 11. Effects of additive concentration on contact angle with different additive.

and porosity changed, different kinds of additives and its concentration influenced membrane performance in different degree.

3.6. Application of PVDF/PPSU blending membrane

Esterification wastewater has some features such as corrosiveness, irritation, toxicity and exorbitant concentration COD_{cr}; if it is treated by MBR module directly, it would cause microbe lose activity and even die, so some treating process (pretreatment – anaerobic/aerobic treatment – advanced oxidation) are adopted to treat esterification wastewater used in this experiment before it was flowed into MBR reactor.

By comparing different optimal membranes in terms of water flux, rejection and contact angle, the blending membrane which added 1% PEG-2000 at PVDF:PPSU was 5:5 and polymer concentration was 19% had the best performance

Table 3
The removal of COD_{cr} by optimal membrane

COD_{cr} (wastewater) (mg L^{-1})	COD_{cr} (treated water) (mg L^{-1})	COD_{cr} removal rate (%)
300	47	84.3
310	49	84.1
360	59	83.6
480	87	81.8
740	135	81.7
800	183	77.1

and were applied in MBR module to treat esterification wastewater (pre-processed); hydraulic retention time was 24 h, aeration intensity was 0.1 $m^3 h^{-1}$, suction time and stopping time were 8, 2 min, respectively. The MBR module operated for a month after activated sludge grew steadily at room temperature. Treatment result was shown in Table 3; it can be seen that with COD_{cr} of wastewater rose, the COD_{cr} removal rate decreased. It indicated that the optimal membrane had a certain treating load, and the COD_{cr} removal rate maintained more than 80% to esterification wastewater with COD_{cr} < 700 mg L^{-1}. However, a steady treatment efficiency was obtained, and the optimal membrane had appropriate pore diameter and great membrane performance.

4. Conclusions

PVDF/PPSU blending membranes were prepared via a phase inversion method. The results of orthogonal polar difference analysis and membrane performance analysis illustrated that (1) when additive were LiCl and $MgCl_2$, polymer concentration is the main factor affecting water flux, rejection and contact angle. When additives were PEG-2000, as for water flux and rejection, polymer concentration is the main factor but for contact angle, the concentration of PEG-2000 is the main factor. (2) The optimal combinations (PVDF/PPSU blend ratio, polymer concentration and additives concentration) were 5:5/17% polymer/1.6% LiCl (wt.%), 7:3/19% polymer/1.0% $MgCl_2$ (wt.%) and 5:5/19% polymer/1% PEG-2000 (wt.%). (3) Larger and longer macrovoids of membrane inner structure were found by observing morphologies of optimal membranes, and the permeability and hydrophilicity of the membrane were improved. (4) Esterification wastewater treatment was conducted by the submerged MBR with the blending membrane which added PEG-2000, and steady treatment efficiency was obtained, the COD_{cr} removal rate maintained 75% or more, the highest COD_{cr} removal rate was 84.3%.

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