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Preparation of Polyvinylchloride Nanofiltration Membrane: Investigation of the Effect of Thickness, Prior Evaporation Time and Addition of Polyethylenglchol as Additive on Membrane Performance and Properties

E. Bagheripour*, A. Moghadassi, S. M. Hosseini

Department of Chemical Engineering, Faculty of Engineering, Arak University, Iran

PAPER INFO	ABSTRACT
	In this work, polyvinylchloride (PVC) membrane was prepared via casting solution technique and phase inversion method. N, N dimethylacetamide (DMAC) was used as primary solvent and tetrahydrofuran (THF) was used as a co-solvent. The effects of parameters such as membrane thickness, evaporation time of casting film before immersion precipitation and addition of polyethylenglchol (PEG) on PVC membrane were studied in terms of deionized water flux (DIW), tensile strength, phase inversion time in non-solvent, water content, permeability flux, salt rejection and antifouling properties.The results revealed that DIW decreased with increasing of membrane thickness and prior evaporation time of casting film. The phase inversion time increased with increase of membrane thickness and prior evaporation time. Tensile strength of fabricated membranes increased sharply while evaporation time was increased. The results showed the reduction of water content percent with increase of casting film evaporation time. Addition of PEG from 1 to 4 % wt improved flux sharply from 1.04 to 13.62 (L/m^2.h). Rejection results showed reducing trend of salt rejection for fabricated membrane with different concentrations of PEG, except at the concentration of 3 % wt PEG. The results clearly showed more stable permeation flux during filtration process for blend PVC/PEG membranes compared to PVC one. *doi: 10.5829/idosi.ije.2016.29.03c.01*

1. INTRODUCTION

Nowadays, membranes have become essential materials not only in industries but also in daily human life.

Preparing inexpensive membranes with specially adapted physicochemical characteristics may be a vital step in future chemical and waste treatment application.

Nanofiltration (NF) is a pressure-driven separation process with membrane pore size in the range of about 0.5–2.0 nm in diameter. The major separation mechanisms of NF involve not only a steric (size exclusion) effect but also an electrostatic partitioning interaction (Donnan exclusion) between the membrane and an external solution [1-4].

Corresponding Author's Email: Bagheripoor_ehsan@yahoo.com (E. Bagheripour)

The NF membranes are increasingly recognized as best processes for water treatment and production of portable water [5]. Most of commercial nanofiltration membranes have been made of polymer. Organic polymer has many advantages such as extensive preparation sources, convenience of manufacturing, low cost, and ease of industrialization [6]. Some polymers such as cellulose acetate (CA), polysulfone (PSf), polyacrylonitrile (PAN), poly vinyli denedifluoride (PVDF), polyetherimide (PEI) and polyethersulfone (PES) could be used to prepare nanofiltration and ultrafiltration membranes by phase inversion method [7-10]. Poly (vinylchloride) (PVC) is an outstanding membrane material because of its stiffness, low cost, excellent physical and chemical properties, acids, alkalis and solvent resistance, as well as mechanical properties [11-14]. Some researches have focused on fabrication of ultrafiltration and microfiltration membranes by phase

inversion method with PVC. Xu and his coworker have studied on fabrication and investigation of PVC/additives hollow fiber ultrafiltration membranes [15]. Hiroshi et al. investigated some factors such as casting solution compositions, polymer concentrations, variety of solvents (N, N-dimethylacetamide (DMAc), DMF and tetrahydrofuran (THF)) and different additives (water, methanol, ethanol and n-propanol) on the flat PVC membranes [16]. However, studies on parameters such as membrane thickness, evaporation time before immersion precipitation and also addition of PEG as an additive on the flat sheet PVC membrane performance and properties seldom is seen. In this research, the effects of PVC membrane thickness, evaporation time of casting film before immersion in non-solvent and different concentrations of PEG as an additive on the properties and performance of PVC membranes were studied. The fabricated membranes were analyzed in terms of deionized water flux, phase inversion time, tensile strength, water content, permeability flux, and salt rejection. The obtained results can be valuable for membrane processes especially nanofiltration process in water recovery and waste water treatment.

2. EXPERIMENTAL

2. 1. Experimental Details   The performance of prepared membranes was studied using the NF dead-end cell (Figure 1). A nanofiltration dead-end cell with a diameter of 4.5 cm and effective filtration area of 11.94 cm^2 was used for filtration experiments. Nitrogen gas at fixed pressure of 6 bar was used as driving-force. The permeation flux was determined by measuring passed water through the membranes in term of L/m^{2}h.

2. 2. Materials   The specifications of used materials are shown in Table 1.

2. 3. Fabrication of Membranes   In this research, membranes were prepared by casting solution technique and phase inversion method.

TABLE 1. Specification of used materials

Materials	Role	Supplied from
PVCgrade S-7054	Basic polymer	BIPC, Iran
DMAC	Primary solvent	Merck
THF	Co-solvent	Dae-Jung
PEG grade 400	Additive	BIPC, Iran
Deionized water (DIW) Na2So4	Feed solution and non-solvent salty feed solution	Chemical engineering lab of Arak university

TABLE 2. Composition of casting solutions (with constant DMAC:THF ratio 85:15)

Membranes No.	PVC % wt	Thickness (μm)	Prior evaporation time (s)	PEG concentration % wt.
1	12	100	60	0
2	12	150	60	0
3	12	200	60	0
4	12	250	60	0
5	12	300	60	0
6	12	150	0	0
7	12	150	15	0
8	12	150	30	0
9	12	150	45	0
10	12	150	70	0
11	12	150	60	1
12	12	150	60	2
13	12	150	60	3
14	12	150	60	4

The preparation proceeded by dissolving the polymer binder (PVC) into the mixed solvents of DMAc and THF with constant ratio of 85:15 [17] in glass reactors equipped with a mechanical stirrer (model: VELP Scientific-Multi stirrer) for more than 6 h. The mixtures were then cast onto clean and dry glass plates at ambient temperature with different thicknesses (100-150-200-250-300 μm) and evaporation times and then were dipped in deionized water (as non-solvent) for exchanging of solvent and non-solvent. The fabricated membranes were transferred to fresh deionized water for 1 day to extract solvent reminded in their structures.   In the next step, different concentrations of PEG were blended with PVC and then prepared mixtures were cast with constant thickness and constant evaporation time (150 μm and 60 s, respectively). The specification and composition of fabricated membranes are shown in Table 2.

2. 4. Membranes Characterization

2. 4. 1. Deionized Water Flux (DIW) Measurements
Circular membranes with top layer contacting the feed solution were placed into the cell. PWF and permeability flux were measured by permeated water collected into a reservoir at 4 bar as driving force and calculated by the following equation [18, 19]:

$$DIW= Q/A \ (\Delta t) \tag{1}$$

where DIW is water flux (L/m^2.h), Q is quantity of permeate (L), A is membrane area (m^2) and Δt is sampling time (h). For permeation flux measurement the above equation was used, too. The salt rejection is defined as followed:

$$Rejection \ \% =1-(C_p/C_f) \tag{2}$$

For which c_p is the concentration of salt inpermeates and c_f is the concentration in the feed that were

measured by conductivity meter (Ohaus Corporation, S/N B143385306, USA) [20, 21].

2. 4. 2. Water Content Experiment
The water content was measured as the weight difference between the dried and swollen membranes.

The membranes were immersed in distilled water for 24 h at ambient temperature (24±2 °C), and following discharge, its surface was wiped by filter paper and weighed (OHAUS, Pioneer, readability: 10^{-4} g, OHAUS Corp., USA). The wet membranes were dried at fixed temperature (50 °C) for 4 h until constant weight was obtained as dry membrane. The following equation can be used in the water content calculation [22, 23]:

$$\%\text{Water content}=((w_w-w_d)/w_w)\times 100 \qquad (3)$$

where w_w and w_d are wet and dry samples weight (g), respectively. For preventing of errors in water content measurements, the experiments were done three times.

2. 4. 3. Mechanical Properties
The tensile strength of fabricated membranes was measured according to ASTM1922-03 standard, reported elsewhere [19, 21, 24].

2. 4. 4. Phase Inversion Time
For analyzing of the effect of casting film thickness, evaporation time and PEG addition into the casting film on the membranes flux and tensile strength (with the knowledge that increasing of phase inversion time in coagulation bath means delayed demixing and it concludes to the formation of membrane with denser structure and decreasing of phase inversion time means instantaneous demixing that leads to the formation of membranes with more porous structure [25]), the phase inversion time of casting film in coagulation bath (non-solvent) was measured by a timer. The phase inversion time was measured between dipping casting film and completing membranes formation.

3. RESULTS AND DISCUSSION

3. 1. Influence of the Casting Film Thickness
In this section, the membranes with constant PVC concentration (12 % wt), constant solvent mixing ratio between DMAC: THF (85:15) and constant evaporation time before immersion of casting film into the non-solvent (60 s) and varied casting film thickness between 100 to 300 µm were fabricated to see the effect of membrane thickness on DIW flux. The DIW flux of the resulting membranes are summarized in Figure 2. The results show a clear trend towards lower DIW flux as the casting film thickness is increased. This is an expected phenomenon in membrane performance. An

increase in membrane thickness leads to decrease in flux. Increasing of casting film thickness, makes more resistance for water to pass through the membranes. In another word, while membrane thickness increases, water should diffuse in a longer path to pass through the membrane. Thus DIW flux reduces by increasing casting film thickness. In addition, the results presented in Figure 3, introduces the effect of casting film thickness on phase inversion time in non-solvent. As it can be seen in this figure, increasing of casting film thickness, leads to the increase of phase inversion time. With higher thickness of casting film, there is more polymeric solution on the glass plate contacting with non-solvent. In this condition, formation of membranes during phase inversion process takes place at longer time and phase inversion time increases.
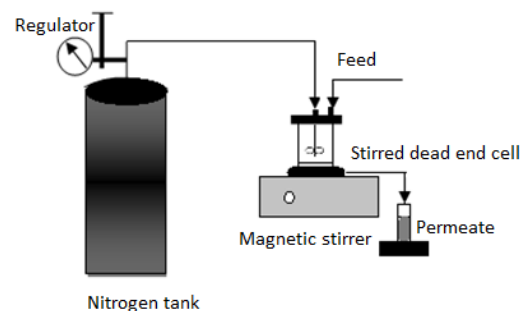

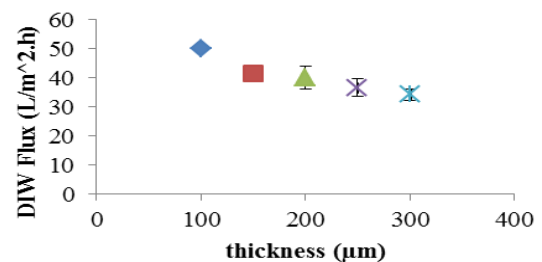**Figure 1.** Schematic diagram of membrane test setup

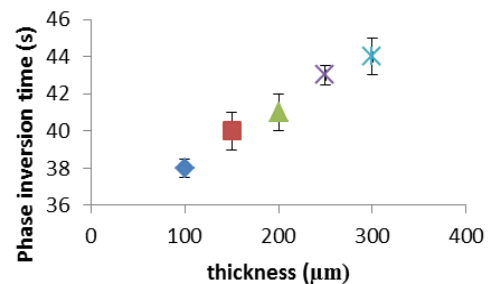**Figure 2.** The effect of membrane thickness on DIW flux

Figure 3. The effect of membrane thickness on phase inversion time
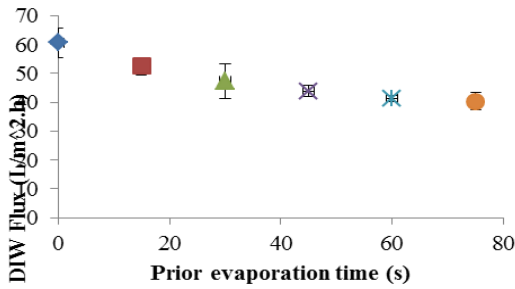
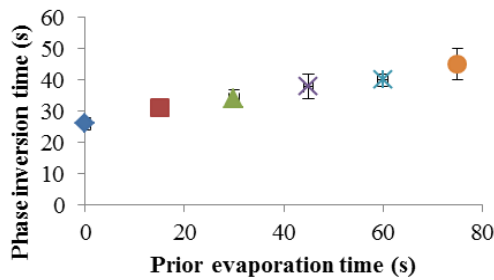**Figure 4.** The effect of prior evaporation time on DIW flux

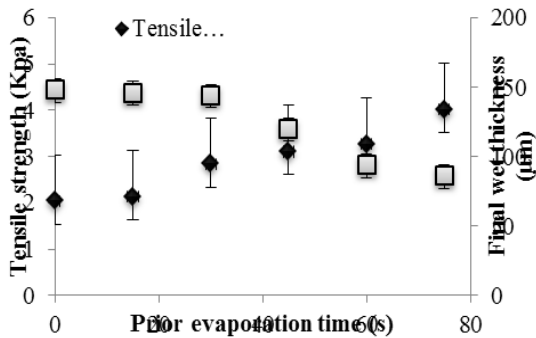**Figure 5.** The effect of prior evaporation time of casting film on phase inversion time

Figure 6. The effect of prior evaporation time of casting film on tensile strength and final wet thickness
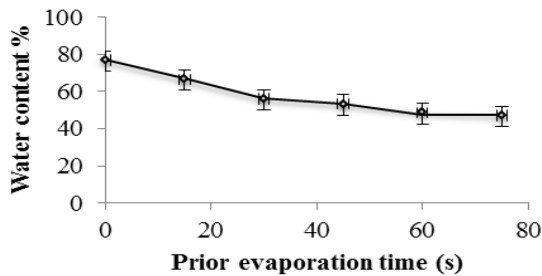

Figure 7. The effect of prior evaporation time of casting film on water content percentage

3. 2. Influence of Various Evaporation Times before Immersion Precipitation   Six membranes

were fabricated with different evaporation times from zero (immediately precipitation in non-solvent) up to 75 s with constant PVC concentration, mixing solvent ratio and casting film thickness (12 % wt, 85:15 and 150 μm, respectively). The permeance of DIW flux versus different evaporation times of casting film is reported in Figure 4. As can be seen, the trend of DIW flux versus increasing evaporation time before immersion step is reducing. When a volatile co-solvent (THF) is used in the casting solution, more evaporation time has significant effect on porosity and flux. In another word, with greater evaporation time, during the evaporation step, the surface of the casting film loses volatile co-solvent (THF). Consequently, the polymer concentration increases in the top layer of the casting film [26]. Higher polymer concentrations in casting film surface leads to the slower diffusion of non-solvent in casting film during phase inversion phenomenon due to higher viscosity of top layer of casting film. Therefore skin layer of fabricated membranes becomes denser that is an expected phenomenon in phase inversion process and during membrane formation. In this case, macrovoides in membrane surface become smaller and flux reduces [27, 28].

The results presented in Figure 5 show the effect of evaporation time of casting film before immersion in coagulation bath on phase inversion time. These results indicate that phase inversion time in coagulation bath increases by increase of evaporation time. It means that evaporation of volatile solvent (THF) in environment before immersion step, slows exchanging of solvent and non-solvent due to increase of polymer concentration and casting film viscosity in the surface of casted film which was mentioned above.

For study on the effect of increasing evaporation time before immersion step on mechanical properties, the tensile strength experiment was performed. The results are shown in Figure 6. The results revealed that the tensile strength of fabricated membranes increased sharply as evaporation time increased. As mentioned before, increase of evaporation time prior immersion of casting film into the coagulation bath, leads to increasing polymer concentration in the top surface of casted film leading to formation of a denser top layer. While evaporation time increases, thickness of dense top layer increases. The increase of dense top layer thickness for membranes containing volatile solvent (THF) by increase of evaporation time is accepted as a result of increase of prior evaporation time [26]. Thus it can be concluded that by increase of dense top layer thickness, the sub-layer (porous support layer) thickness reduces. Consequently, tensile of fabricated membranes versus its strength and breaking point increases.

In addition the final wet thickness of fabricated membranes after phase inversion step was measured by a digital micrometer. For all of the fabricated membranes, this experiment was done three times and

the results are average of measurements. These results as presented in Figure 6 indicate that membranes fabricated with more evaporation time before immersion step have lower thickness after completion of phase inversion process due to exchange of porous layer with dense layer. These results have a good agreement with the results reported about tensile strength. It is obvious that as membrane's structure turns to the denser structure, its resistance against strength increases. Besides, for more study on membrane structure prepared with different casting film evaporation time, water content was also measured by Equation (3). These results as summarized in Figure 7 showed reduction of water content percentage with increase of casting film evaporation time (before immersion precipitation in non-solvent). These results indicate reduction of macrovoides in membrane structure and reduction of membrane porosity by increase of casting film evaporation time. The results are in agreement with reduction of DIW flux, increase of phase inversion time and tensile strength and reduction of final wet thickness which were explained before.

3. 3. Influence of Various Concentrations of PEG
Figure 8 indicates the effect of PEG addition with different concentrations into the casting solution on membrane performances (DIW flux, permeance flux and salt rejection). Comparison of results shows increasing of DIW flux and permeance flux by increase of PEG concentration into the casting solution. Addition of PEG from 1 to 3 % wt improved flux with the soft slop. But addition of PEG at concentration of 4 % wt had the most effect on flux. In fact, addition of PEG with 4 % wt into the casting solution increased flux significantly compared to pristine membrane without PEG.

The velocity of the demixing process (delay or instantaneous) affects the morphology of the membranes. Delay demixing almost always leads to the formation of a dense structure for membranes. Whereas, instantaneous demixing with faster exchange of solvent and non-solvent and phase inversion in coagulation bath happens after lower period of time. Consequently, contrary to delayed demixing, formation of macrovoids increases. Thus, more porous structure membranes is synthesized [29].
Supply of PEG as additive into the casting solution, due to having the same properties with non-solvent (deionized water), increases the unstability of casting solution. More unstability of casting solution leads to easier and faster diffusion of solvent from casting film into the non-solvent [30]. Besides, addition of PEG with the same properties to deionized water into the casting solution leads to the adsorption of deionized water in

coagulation bath with higher rates, during phase inversion process. Thus, phase inversion process occurs at higher rate and velocity. This type of behavior leads to instantaneous demixing. The results of measured phase inversion time in coagulation bath are presented in Figure 9 which confirm the above expression about occurring of instantaneous demixing as a result of presence of PEG in the casting solution. In this situation formation of macrovoides in the membrane structure increases which was observed in the literatures [31, 32]. Therefore, flux increases .
As seen in Figure 8 (results of salt rejection), there is a reducing trend for salt rejection of fabricated membranes with different concentrations of PEG, except at concentration of 3 % wt PEG. As mentioned about increasing of macrovoides due to presence of PEG in casting solution, the reduction of salt rejection was expected. In fact, when macrovoides grow in membrane' s structure/surface, rejection decreases [33]. In another point of view, presence of PEG in the membrane structure leads to more adsorption of water on membrane surface during filtration experiment. This phenomenon prevents accumulation/adsorption of salts on membrane surface. In this situation, salt rejection increases. In fact, it can be said, supplying of materials such as PEG with same properties to non-solvent (deionized water) as additive has dual effect on membrane performance (salt rejection). In reality, the salt rejection of the fabricated membranes depends on superiority of the both phenomena which was mentioned earlier. It means, increase of salt rejection in membrane containing 3 % wt PEG can be related to the superiority of more water adsorption on membrane surface compared to growing of membrane mocrovoides.

In addition for better analysis of the structure of fabricated blend PVC/PEG membranes, final wet thickness of fabricated membranes was also measured. These measurements are shown in Figure 10. The results confirmed the increase of fabricated membranes porosity by addition of PEG. While PEG concentration increases in the casting solution, the membrane porosity also increases.
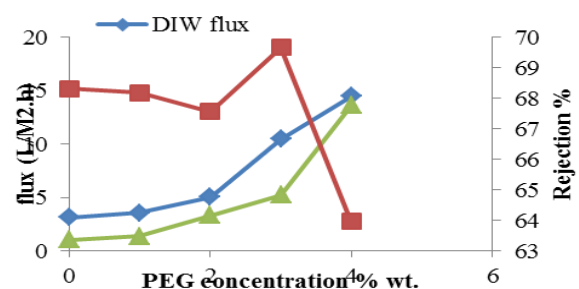

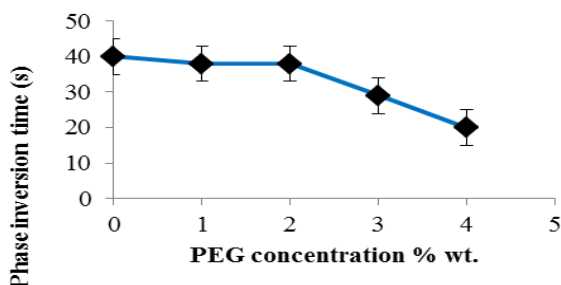**Figure 8.** The effect of PEG concentration on membrane performance

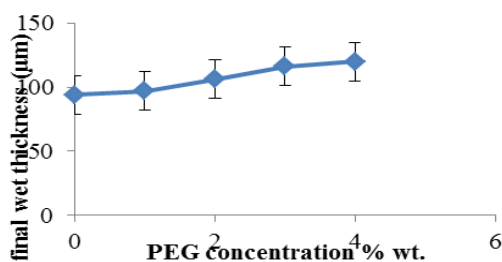**Figure 9.** The effect of PEG concentration on phase inversion time

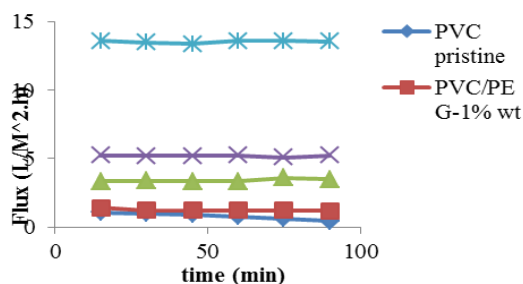**Figure 10.** The effect of PEG concentration on final wet thickness of membrane

Figure 11. The effect of PEG concentration on foling

3. 4. Influence of PEG on Antifouling Properties

The variation of permeate flux with time for membranes during filtration process is presented in Figure 11. The permeability and selectivity of membranes are mainly controlled by the hydrophilicity and structure of membranes. As can be seen in the figure, the permeability flux of membranes decreased by increase of processing time that may be related to the formation of cake layer on membranes' surface [34]. The results clearly show that the fabricated blend PVC/PEG membranes have more stable permeation flux during filtration process. In reality, presence of PEG as additive in the casting solution, due to having same properties as water, increaseses membrane hydrophilicity [35] which can decrease association and precipitation/adsorption of solutes in water onto the membrane surface during filtration experiment leading to the higher and more uniform flux of blend

membranes compared to pristine hydrophobic PVC. Thus, formation of cake layer on membrane surface reduces. In this case, decline of flux during filtration experiment decreases.

4. CONCLUSION

The current research was focused on the effect of membrane thickness, evaporation time of casting film in air before immersion precipitation and addition of different concentrations of PEG as additive to the casting solution, on PVC membrane performance and structure. The fabricated membranes were analyzed in terms of DIW flux, phase inversion time, tensile strength, water content, final wet thickness, salt rejection and antifouling properties. The results revealed that DIW decreased with increase of membrane thickness and prior evaporation time of casting film. The phase inversion time increased with increase of membrane thickness and prior evaporation time. Tensile strength of fabricated membranes increased sharply while evaporation time was increased. The results showed reduction of water content percentage with increase of casting film evaporation time. Addition of PEG from 1 to 4 % wt improved flux sharply from 1.04 to 13.62 $(L/m^2.h)$. Rejection results indicated reducing trend of salt rejection for fabricated membrane with different concentrations of PEG, except at the concentration of 3 % wt PEG. The results clearly showed more stable permeation flux during filtration process for blend PVC/PEG membranes compared to PVC one.

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E. Bagheripour, A. Moghadassi, S. M. Hosseini

Department of Chemical Engineering, Faculty of Engineering, Arak University, Iran

چکیده

در این تحقیق، غشای از جنس پلی وینیل کلراید با استفاده از تکنیک محلول پلیمری و روش وارونگی فازی تهیه گردید. دی متیل استامید به عنوان حلال اولیه و تتراهیدرو فوران به عنوان کمک حلال استفاده شد. اثرات پارامترهایی چون ضخامت غشا، زمان تبخیر فیلم پلیمری پیش از مرحله غوطه وری و همچنین اثر افزودن پلی اتیلن گلایکول به عنوان افزودنی بر غشای پلی وینیل کلراید با اندازه گیری شار آب بدون یون، مقاومت کششی، زمان وارونگی فاز در حمام غیر حلال، آب همراه، شار آب عبوری، پس دهی نمک، و خواص ضد گرفتگی مطالعه شدند. نتایج نشان دادند که شار آب خالص با افزایش ضخامت غشا و افزایش زمان تبخیر فیلم پلیمری، کاهش میابد. زمان تغییر فاز با افزایش ضخامت و زمان تبخیر فیلم پلیمری افزایش یافت. مقاومت مکانیکی غشاهای ساخته شده با افزایش زمان تبخیر فیلم پلیمری شدیدا افزایش یافت. نتایج نشان دادند که افزودن پلی اتیلن گلایکول از غلظت ۱ تا ٤ درصد وزنی باعث بهبود شار آب از مقدار ۱/۰٤ تا مقدار ۱۳/٦۲ شد. نتایج پس دهی نشان دادند که مقدار جداسازی نمک برای غشاهای تهیه شده با غلظتهای مختلف از پلی اتیلن گلایکول روندی رو به کاهش داشت به غیر از غلظت ۳ درصد وزنی از پلی اتیلن گلایکول ک این غلظت باعث باهبود مقدار جداسازی نمک گردید. به علاوه نتایج این آزمایش بیانگر شار عبوری پایدارتر غشاهای ترکیبی پلی وینیل کلراید/ پلی اتیلن گلایکول در برابر گرفتگی در مقایسه با غشای پلی وینیل کلراید خالص بود

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