



Fabrication of (Acrylonitrile Butadiene Styrene/Poly Ethylene Glycol) Nanofiltration Membrane: the Effect of PEG Concentration and Operating Conditions on Membrane Performance

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ABSTRACT

In the current research, ABS-co-PEG nanofiltration membrane was prepared by solution casting technique using N, N dimethyl acetamide as solvent. The effect of PEG concentration as additive in the casting solution on membrane flux, salt rejection, phase inversion time, water content, membrane porosity, membrane tensile strength and fouling was studied. Also the effect of operating conditions such as feed concentration, pressure and temperature on membrane performance were also studied. It was found that increase of PEG content up to 6 %wt in the casting solution initially led to increase in flux and decrease of salt rejection in prepared membranes. The flux was decreased and salt rejection increased by more increase in PEG content from 6 to 10 %wt. In addition, presence of PEG in membrane structure caused to formation of more stable flux during filtration time against fouling. Increase of feed salt concentration caused to flux decreasing. The ABS/PEG membrane showed more stable flux against increase of feed concentration. Moreover, flux was increased by increase of operating pressure and feed temperature. The results also showed a clear trend towards higher values of tensile strength by increase of PEG content ratio.

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1. INTRODUCTION

Nowadays, nanofiltration (NF) membranes are widely utilized as active separators because of its advantages such as low-operation pressure, high-permeate flux, and high retention of multivalent ion salts. The NF process has been used in many applications such as wastewater reclamation, industrial water production, water softening and in the separation of compounds having different molecular weights [1-6]. Different materials can be used for membrane fabrication such as metals, ceramics, graphite, glass, and polymers. Most commercially available nanofiltration membranes are made of polymer. In fact, all polymers can be used as barrier or membrane materials, but their chemical and physical properties specifications have limited number

are used in practice [7]. The organic polymer has many advantages, such as extensive sources, convenient manufacture, low cost, and easy to achieve the industrialization, but it also has the disadvantage to overcome, such as bad thermo stability, weak anti-fouling and poor anti-swelling etc. There are some extensive polymers such as cellulose acetate (CA), polysulfone (PSf), polyacrylonitrile (PAN), polyvinylidenedifluoride (PVDF), polyetherimide (PEI) and polyethersulfone (PES) could be used to prepare nanofiltration and ultrafiltration membranes by phase inversion method [8-11]. Acrylonitrile butadiene styrene (ABS) is a new copolymer with good filtration characteristics [12]. ABS is a commercial material with relatively low cost and good balance of mechanical properties and moderate glass transition temperature (110 °C). This copolymer is a mixture of styrene, butadiene and acrylonitrile. Styrene and butadiene are hydrophobic and acrylonitrile has a hydrophilic nature [12]. The hydrophilic, electronegative and polar

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properties of PEG and the mobility and flexibility of the PEG chains also makes nominate this polymer as modifier in membrane matrix. Moreover the accessibility, non-toxic and low cost of PEG motivates the PEG selection for the aim. Presence of PEG in casting solution can increase thermodynamic instability of the cast film and consequently causes instantaneous demixing in the coagulation bath. This produces an asymmetric membrane with macro voids in the sub layer [13, 14]. Some researches have been studied the effect of PEG on morphology and performance of polymeric membranes. Mohammadi and his coworkers studied PEG-CA asymmetric membranes [15]. In another research, the effect of molecular weight of PEG on PSf membrane morphology and transport properties was studied [16]. Also the effect of PEG on gas permeation membranes was investigated for capturing carbon dioxide from CO₂/N₂ Streams [17]. However, studies on the fabrication and characterization of blend ABS/PEG nanofiltration membrane in application of water treatment and purification was not seen yet. Hence, the effect of PEG concentration on ABS NF membrane performance was investigated. The fabricated membranes were characterized in terms of DIW (deionized water) and permeation flux, salt rejection, phase inversion time, tensile strength, water content, porosity and their ability against fouling. Besides, some parameters such as effect of feed temperature, concentration and operation pressure on flux were analyzed.

2. EXPERIMENTAL

2.1. Materials The details of supplied materials are shown in Table 1.

2.2. Fabrication of the Membranes Different compositions of ABS/PEG/DMAC with constant concentration of ABS (20 wt%) as basic polymer were prepared by stirring it for 4 hours. Then, the solutions were degassed in an ultrasonic cleaner bath for about 2 hours at 20°C (Parsonic, IRAN) to remove air bubbles. After that, the solutions were casted on smooth glass plates by a film applicator with constant thickness of 200 μm.

TABLE 1. Details of used materials

Materials	Role	Supplied from
ABS	Basic polymer	BIPC, Iran
DMAC	solvent	Merck
PEG grade 400	additive	BIPC, Iran
Deionized water Na ₂ SO ₄	non-solvent Feed solution	Merck

The glass plates were then immersed in deionized water bath at ambient temperature (20 °C) for immediate phase inversion process. The membranes were kept in fresh deionized water for one day to extract reminded solvent in their matrix. Their compositions are presented in Table 2.

2.3. Phase Inversion Time Measurement The phase inversion time in non-solvent bath was measured by a digital chronometer. The time was between the immersion of casting film in non-solvent bath and the floating of solidified film in the bath.

2.4. Membranes Characterization

2.4.1. DIW Flux, Permeation Flux and Rejection Measurements

The DIW flux, permeation flux and rejection measurements were carried out in a dead-end cell of nanofiltration membrane with a diameter of 4.5 cm and effective membrane area of 11.94 cm². All experiment were done at room temperature. The schematic diagram of setup is shown in Figure 1. The circular samples were placed into the cell with top layer in contact feed. The cell reservoir was filled with deionized water and DIW flux test was done. Then it refilled with salt solute (1000 ppm sodium sulfate solution) for permeation flux and salt rejection experiments. Nitrogen gas by 4 bars was used as driving-force of nanofiltration membrane experimental. The below equations were used for DIW, permeation and salt rejection calculations respectively.

TABLE 2. Composition of various casting solutions

Membranes (No.)	ABS (wt %)	PEG (wt %)	DMAC (wt %)
1	20	0	80
2	20	2	78
3	20	4	76
4	20	6	74
5	20	8	72
6	20	10	70

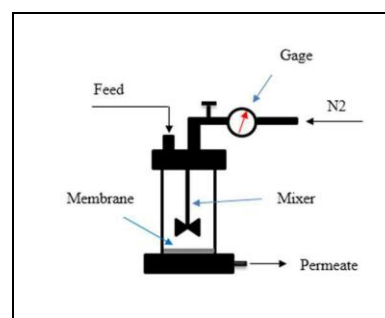


Figure 1. The nanofiltration experimental setup

$$\text{Flux} = \left(\frac{Q}{A \cdot \Delta t} \right) \quad (1)$$

Where flux is deionized and permeation flux (L/m²h), Q is quantity of permeate (L), A is membrane area (m²) and Δt is sampling time (h) [18].

$$\text{Rejection\%} = \left(\frac{C_f - C_p}{C_f} \right) * 100 \quad (2)$$

For which C_p is the concentration of salt in permeates and C_f the concentration in the feed that they were measured by conductivity meter (Ohaus, U.S.A.) [19].

2. 4. 2. Water Content/Porosity For water content measurements, two weights of wet dried membranes were measured by a digital scale (OHAUS, Pioneer TM, Readability: 10⁻⁴ g, OHAUS Corp). For wet membranes, they were kept in deionized water one day and for dry membranes, they were placed in heating oven at 60 degrees of centigrade for one day too. Below expression was used for water content calculations:

$$\text{Water content\%} = \left(\frac{W_w - W_d}{W_w} \right) * 100 \quad (3)$$

In addition to calculate the fabricated membranes porosity, the following equation was used:

$$\text{Porosity} = \left(\frac{W_w - W_d}{\rho_f V_m} \right) * 100 \quad (4)$$

where, ρ_f and V_m are water density (g/cm³) and membrane pieces volume (cm³) [20].

2. 4. 3. Membranes Mechanical Property For study on the effect of PEG concentration on ABS NF membranes mechanical properties, tensile strength of prepared membranes was measured. Measurements of mechanical properties were conducted according to ASTM1922-03 standard that was reported elsewhere [21].

3. RESULTS AND DISCUSSIONS

3. 1. Effect of PEG Concentration on Membrane Morphology and Mechanical Property

3. 1. 1. Phase Inversion Time For determination of the effects of PEG concentration on morphology and structure of the prepared membranes, phase inversion time of casting film in non-solvent was measured. The rate of the demixing process affects the membrane morphology. Decreasing of phase inversion time during formation of membranes in non-solvent means higher rate of exchanging of solvent and non-solvent and consequently, results in instantaneous demixing

phenomenon and formation of macro voids for the prepared membranes [22, 23]. Inversely, increasing of phase inversion time, leads to demixing phenomenon which makes a membrane with dense structure [22, 23]. Thus, it can determine the morphology of the fabricated membranes by measuring of phase inversion time of casting film in coagulation bath. There are data of measured phase inversion time of casting film versus of PEG concentration in Figure 2. As it can see in this figure, the phase inversion time of casting film has two types of behavior while PEG concentration was increased in casting solution. It is obvious, increasing of PEG concentration from 0 to 6 wt%, had led to decreasing of phase inversion time in coagulation bath. In another word, it had led to instantaneous demixing. By increasing of more PEG concentration up to 6 to 10 wt%, phase inversion time has increased. Presence of PEG as additive in casting solution, due to involving the same properties with non-solvent (deionized water) increases instability of casting solution. More instability of casting solution increases solvent potential to leave the casting film and diffuses in to non-solvent happens with more rates. In addition, PEG in casting film due to its hydrophilic properties, adsorbs non-solvent (deionized water) with more rates too. Above phenomenon's leads to instantaneous demixing. Thus, the phase inversion time in coagulation bath decreases by increasing of PEG concentration to 6 wt%. The increasing of phase inversion time in coagulation bath by increasing PEG concentration from 6 to 10 wt%, can describe by increasing of casting solution viscosity. Increasing the viscosity of casting film declines the exchange rate of solvent and non-solvent during the phase inversion process and it can leads to delay demixing and consequently formation of a denser structure. In reality, it should be mentioned that addition of hydrophilic additives such as PEG to casting solution has two different effects on membranes structure and performance.

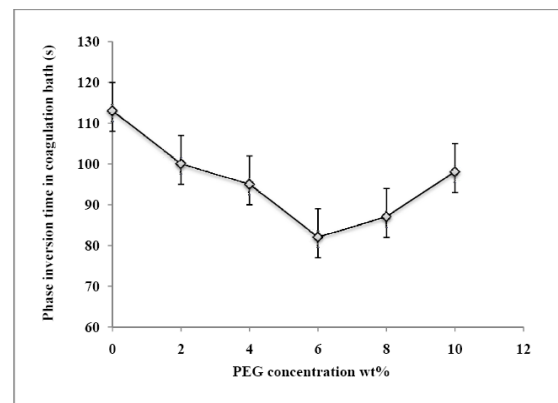


Figure 2. Effect of PEG concentration on phase inversion time in coagulation bath

It means, the final structure of fabricated membranes after addition of PEG is depending on superiority of instantaneous or delay demixing phenomenon [24]. In this research, base on presented data in Figure 2, it seems that by increasing of PEG in the range of 0 to 6 wt%, the preferred phenomenon is instantaneous demixing (lower phase inversion time) and in the range of 6 to 10 wt% PEG, preferred phenomenon has delayed demixing (longer phase inversion time).

3. 1. 2. Water Content and Porosity

Hydrophilicity of the membranes is directly related with % water content [25]. The tests (water content and porosity) were performed for determining the variety of membranes hydrophilicity and porosity by increasing PEG concentration. The influence of PEG concentration on ABS nanofiltration membranes water content and porosity are shown in Figure 3. The hydrophilicity of the blend ABS/PEG membranes fabricated by PEG in the range of 0-6 wt%, is higher than that of ABS membrane. This is because of interface macro voids formation due to PEG presence in the casting solution which gives higher void volume in membranes structure. These macro voids are responsible for the higher water content. This kind of behavior what was seen at other literature is due to instantaneous demixing phenomenon as described before. In this situation, membranes porosity increases (see presented data for membranes porosity in Figure 3). Another reason is that the PEG is a water-soluble polymer, which can also enhance the % water content [26]. Higher PEG concentration to 10 wt% due to increasing casting solution viscosity and concentration, leads to denser structure [27, 28]. The results of porosity versus PEG concentration from 6 to 10 wt% firm this mater. The summarized results for water content and membranes porosity are in agreement with presented data in Figure 2 about phase inversion time (instantaneous or delay demixing).

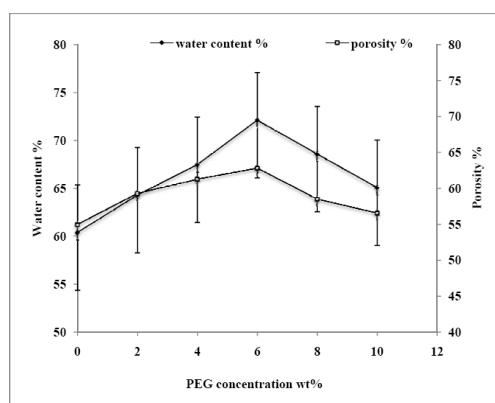


Figure 3. Effect of PEG concentration on water content and membranes porosity

3. 1. 3. Tensile Strength Mechanical properties of fabricated ABS/PEG nanofiltration membranes were measured by tensile strength. The tensile strength results reflect the required energy for breaking membranes against of stretching. Figure 4 indicates the effect of variation PEG concentration (0-10 wt %) on ABS (with constant concentration 20 wt %) tensile strength. The results show a clear trend towards higher values of tensile strength using PEG with different concentrations. It can be concluded that membranes made from blend ABS/PEG are stronger and more flexible than pristine ABS membrane.

This may be due to mobility and flexibility of PEG molecules which placed between the ABS chains in membrane matrix. This makes facile the slipping of ABS chains on together and improves the membrane tensile strength. In the other point of view, increase of casting solution viscosity decreases membrane porosity (see Figure 3, after 6 wt% PEG) which provides a denser structure for the prepared membranes.

3. 2. The Effect of PEG Concentration on Flux and Salt Rejection

DIW flux and permeability flux of the fabricated membranes are presented in Figure 5. These measurements confirm the behaviors observed in the reported data about phase inversion time, water content and porosity in Figures 2 and 3, respectively. While PEG concentration in to the casting solution increases, macro voids formation and porosity in the membranes structure increase by dissolving of PEG from 0-6 wt% (see Figure 3). Therefore, higher flux (DIW and permeation flux) is an expectable phenomenon. However, the macro voids are smaller at higher polymer concentration (PEG 6-10 wt %). This is a known effect for the phase inversion process [15]. Higher polymer concentration (ABS/PEG) in to the casting solution results in higher polymer concentrations at the interface of the casting film and the non-solvent bath (deionized water).

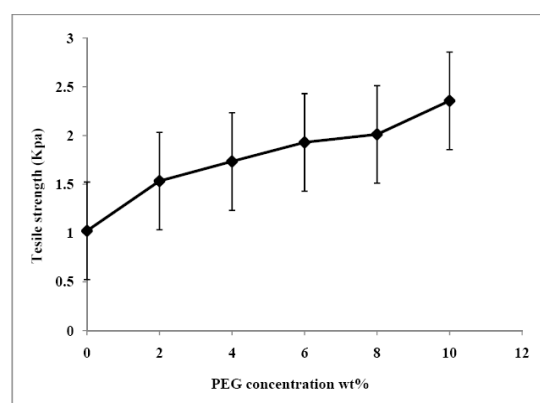


Figure 4. Effect of PEG concentration on blend membranes tensile strength

As the diffusion of non-solvent in to the casting film has slowed down due to the higher viscosity of the casting film, macro voids formation is reduced [29-32].

This type of phenomenon can be the reason of reduction flux after addition PEG up to 6 wt%. This mechanism is anticipated when the concentration of PEG is significant in comparison to the concentration of casting solution. The results of salt rejection summarized in Figure 6 are in agreement with the data's of flux reported in Figure 5, where flux increases, salt rejection decreases and inversely decreasing of flux contains of enhancing of salt rejection.

3. 3. The Effect of PEG on Membranes Fouling

Flux variation of fabricated membranes against of filtration time (min) with different concentration of PEG as additive and constant concentration of ABS (20 wt%) has illustrated in Figure 7. As it is clearly seen in this figure, by increase of PEG concentration in the casting solution, the blend fabricated membranes shows more stable flux during filtration time. This is assigned to hydrophilic characteristic of PEG which improves the ability of membranes in water absorption.

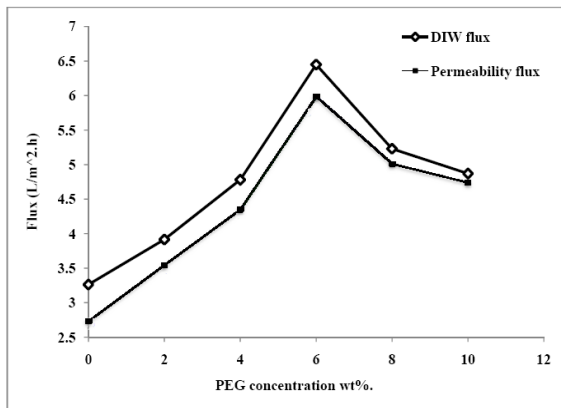


Figure 5. Effect of PEG concentration on DIW and permeation flux

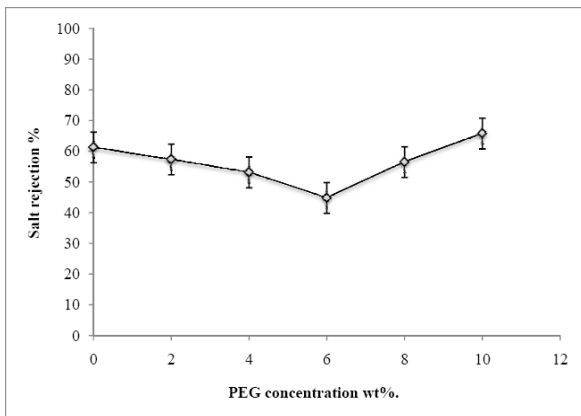


Figure 6. Effect of PEG concentration on salt rejection

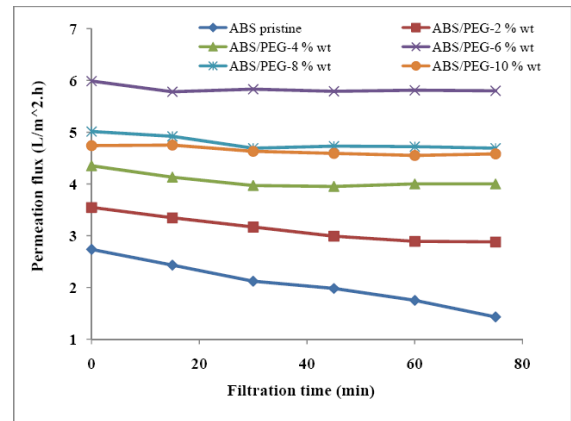


Figure 7. Effect of PEG concentration on ABS membrane anti-fouling properties

In this situation, more water can pass through the membranes which decline the fouling rate for the modified membranes [33].

3. 4. Effect of Concentration on Nanofiltration Permeation Flux

The feed concentration effect experiments were carried out at 4 bars as operating pressure for membranes fabricated pure ABS and blend ABS/PEG- 4wt%. The results are presented in Figure 8. This figure indicates that the permeation flux decreases with increasing feed salt concentration. The decrease in flux is probably attributed to the increasing osmotic pressure. More salt adsorption on the fabricated membrane surface due to increasing feed concentration leads to reduction the real NF driving force of the membranes [34]. But as it is obviously seen in this figure, the blend membrane (ABS/PEG- 4wt%) has more stable flux against of increasing feed concentration that it is because of hydrophilic PEG presence in membrane structure. Although PEG there is in all samples, but it seems that presence of PEG with the concentration of 4 wt% into the casting solution in the optimum.

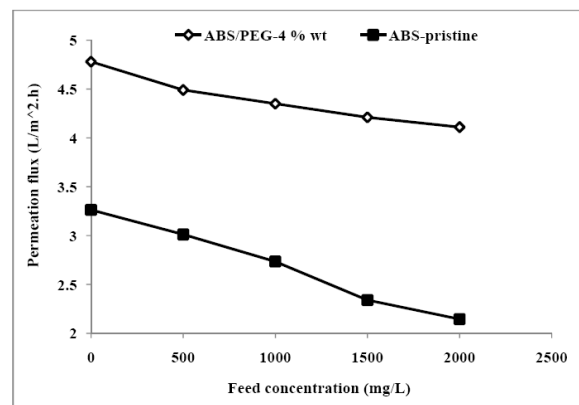


Figure 8. Effect of feed concentration on permeation flux

The lower concentrations less than 4 wt% have affected on the hydrophilicity improvement, but as the concentration of PEG has increased to 4 wt%, its effect on hydrophilicity was increased too because of its concentration. Higher concentration can be affected the hydrophilicity rather than lower ones.

3. 5. The Effect of Applied Pressure on DIW Flux

This experiment was carried out to study the effect of applied pressure (3-5 bars) on DIW flux. When the feed pressure increases from 3-5 bars, DIW flux increases linearly, as it can see in Figure 9. This type of behavior was typical for about most of the NF membranes. As it has mentioned in the Spiegler–Kedem model [35], the water flux of NF membrane is in direct proportion to the trans-membrane pressure that is the difference between operating pressure and osmotic pressure. The osmotic pressure can be eliminated compared to the operating pressure, because of that it is very small when the concentration of salts is low or zero. Therefore, the DIW flux of the fabricated membranes has increased proportionally with the increasing of operating pressure.

3. 6. The Effect of Feed Temperature on DIW Flux

Figure 10 displays the effect of increasing feed temperature on ability of membranes in passing water.

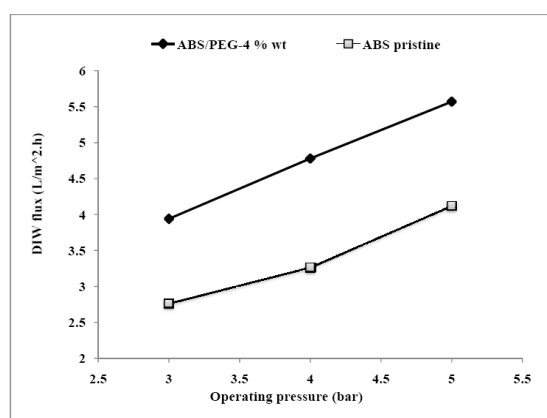


Figure 9. Effect of different operating pressure on DIW flux

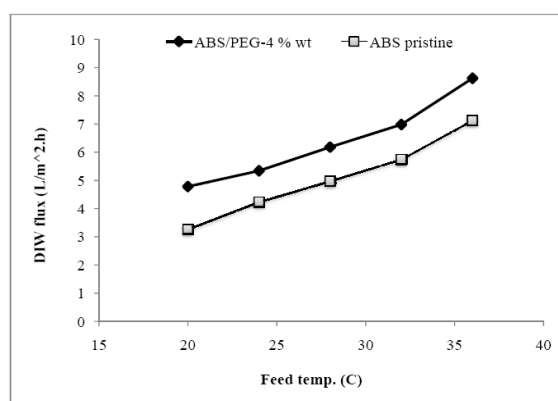


Figure 10. Effect of feed temperature on DIW flux

The trend of the plot shows that the DIW flux of the both pristine ABS and blend ABS/PEG-4 wt% nanofiltration membranes are improving while feed temperature increases. A reason of increase in DIW flux by increasing of feed temperature can be due to the decrease of viscosity of the feed solution with increase in temperature. One more reason can be due to an increase of mass-transfer coefficient by increase of feed temperature [36].

4. CONCLUSION

In the current work nanofiltration membrane was successfully fabricated from ABS/ PEG. The effect of varying concentrations of PEG as additive on ABS membrane performance and structure was studied. By measuring phase inversion time of casting film in coagulation bath, the effect of PEG addition to ABS membrane structure determined by knowing of the phase inversion mechanisms (instantaneous or delay demixing). For more study on membranes structure, water content, porosity and tensile strength were measured. It was concluded that addition of PEG from 0-6 wt% leads to instantaneous demixing phenomenon during phase inversion process that it makes membranes structure more porous. In this case flux increased and salt rejection reduced. In addition, it was concluded that presence PEG up to 6 wt% because of increasing casting solution viscosity and concentration, leads to delay demixing phenomenon that it makes membrane structure denser compared to before. This kind of behavior reduced flux and increased salt rejection. Increase of feed salt concentration caused to flux decreasing. The ABS/PEG membrane showed more stable flux against increase of feed concentration. Moreover, flux was increased by increase of operating pressure and feed temperature. The results also showed a clear trend towards higher values of tensile strength by increase of PEG content ratio.

5. ACKNOWLEDGMENT

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در این پژوهش غشای نانوفیلتراسیون بر پایه ABS/PEG به کمک تکنیک قالب گیری محلول پلیمری و با استفاده از دی متیل استامید به عنوان حلال ساخته شد. اثر غلظت ماده افزودنی پلی اتیلن گلیکول در محلول پلیمری بر فلاکس غشا، جداسازی نمک، سرعت تغییر فاز، محتوای آب و تخلخل غشا، مقاومت مکانیکی و میزان گرفتگی آنها مورد بررسی قرار گرفت. همچنین اثر شرایط عملیاتی از قبیل غلظت خوراک، دما و فشار بر عملکرد غشاها بررسی شد. مشخص شد که افزایش میزان غلظت پلی اتیلن گلیکول تا ۶ درصد وزنی در محلول پلیمری در ابتدا سبب افزایش میزان فلاکس و کاهش میزان جداسازی نمک گردید. با افزایش بیشتر میزان غلظت افزودنی از ۶ تا ۱۰ درصد وزنی مجدداً فلاکس کاهش و میزان پس دهی نمک افزایش یافت. همچنین استفاده از PEG در ساختار غشا سبب ایجاد فلاکس پایدار و کاهش میزان گرفتگی غشا گردید. افزایش غلظت خوراک نیز سبب کاهش میزان فلاکس گردید. غشاهای اصلاح شده حاوی پلی اتیلن گلیکول فلاکس پایدارتری با افزایش میزان غلظت خوراک داشتند. علاوه بر آن افزایش میزان فشار عملیاتی و دمای خوراک سبب بهبود میزان فلاکس در غشاها گردید. نتایج به صورت آشکاری نشان داد که افزایش میزان ماده افزودنی سبب بهبود خاصیت مکانیکی غشاها گردید.

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