

Development and characterization of asymmetric nanofiltration surfactant membranes with different PES concentration

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Abstract In this study a new formulation of dope solution consisting of polyethersulfone, N-methyl-2-pyrrolidone, water and surfactant were formulated from 19 to 27 wt% of PES concentration. The polyvinylpyrrolidone additive (PVP K15) and surfactant (Triton X-100) were added into the dope solution as pore former and to improve the membrane porosity, respectively. Moreover, to fabricate the nanofiltration-surfactant (NFS) membrane. From this study, experimental data showed that the membrane prepared from polymer concentration of 20.42 wt% (optimum) exhibited of high water flux ranging from $6.30 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ to $8.94 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$, $3.26 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ of salt permeation and good NaCl rejection of 43.10%.

Keywords: dope solution ,the membrane porosity ,fabricate the membrane , water flux.

تطوير وتشكيل اسطح الفلاتر النانوية اللامتماثلة باستخدام تراكيز مختلفة من البوليمر PES

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المخلص في هذه الدراسة هناك صيغة جديدة للمحلول تتكون من بولي ايثر سولفن (PES 19-27%) والمذيب N_ميثايل_2_بايروليدون (NMP) و البولي فينال كمادة مضافة (PVP k15) والماء المقطر و (Triton X-100) كمادة سطحية. كل هذه المكونات استخدمت من اجل انتاج محلول مطور من اجل تحسين مسامية الغشاء وبالتالي يتم انتاج غشاء الترشيح الدقيق جدا (NFS). في هذه الدراسة اظهرت البيانات التجريبية للغشاء الذي تم تحضيره من البوليمر ذو التركيز 20.42% (الامثل) اظهرت تدفق المياه بكمة كبيرة تتراوح بين $6.30 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ الى $8.94 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ والاملاح النافذة بمعدل $3.26 \times 10^{-6} \text{ m}^3/\text{m}^2\text{s}$ و افضل معدل لرفض ملح كلوريد الصوديوم (NaCl) 43.10%. هذه اعلى انتقائية لغشاء الترشيح الدقيق جدا و الامتثال تمت بنجاح و التي حققت اعلى اداء للفصل و احدث غشاء نانوي مصنع (NFS) كانت تملك مسامات ذات حجم ضيق وتوزيع الجيد لتلك المسامات والهياكل الغير متماثلة الرائعة . لذلك ومع الاداء العالي لحزم الفصل والتفاصيل الهيكلية الدقيقة ، وايضا قدمت الاغشية النانوية المبتكرة (NFS) امكانات ممتازة نحو تطوير الاغشية ومنصة كبيرة لانتاج اغشية الترشيح الدقيق جدا عالية الاداء انتاجا محليا لمختلف التطبيقات في المستقبل.

الكلمات المفتاحية: محلول مطور، غشاء الترشيح الدقيق جدا، الهياكل الغير متماثلة، حزم الفصل، غشاء نانوي.

Introduction

In order to fabricate high performance membranes[6], the polymer concentration for the casting solution must be sufficiently high in order to produce a membrane with dense surface layer[1] and good mechanical support layer for high separation profile[8]. The new formulations were obtained using turbidimetric titration measurement. The real compositions of the dope solutions and its viscosities were tabulated.[2,13]. It was found that the increasing of polymer concentration required least amount of solvent and non-solvent which resulted to higher viscosities. The variation of polymer concentration in dope solution leads to a different type of membrane structure and exhibited different membrane characteristics. In addition[3,4]. the existing of optimum polymer concentration is needed to be considered[5]. It is shown that dope solution begins to exhibit significant chain

entanglement at a critical/optimum polymer concentration[7,10]

Materials and method

Chemicals and raw materials

Polyethersulfone (purchased from Amoco Chemicals, was used as a membrane material. N-methyl-2-pyrrolidone (NMP) and poly(vinyl pyrrolidone) (PVP) were purchased from Merck, Darmstadt, Germany were used as solvent and additives, respectively, while distilled water was used as a non-solvent. Water used as a coagulation medium. Sodium chloride (NaCl) and multivalent salts that are Na_2SO_4 , MgSO_4 and MgCl_2 were used for nanofiltration performances test[9,11,12].

Membrane preparation and fabrication

The casting solution was prepared by the polymer concentration of 20.5 wt%. Firstly, PES was dried for at least 24 hours in a vacuum oven at a temperature of about $100 \pm 10^\circ\text{C}$ in order to remove

all absorbed water vapor. PES was dissolved at about 56 oC in a multi-component solvent. The polymer solution was put into an ultrasonic bath for about 3 hours to remove the bubbles and then was kept at room temperature for 24 hours. Based on the dry/wet phase inversion technique, membranes were cast using of high precision membranes casting machine. The membranes were cast on a glass plate at ambient temperature and membranes casting condition was fixed at about 10s of casting time, 150 μm of casting thickness and 35.00 of membranes length. Subsequently, the membranes were immersed into an aqueous bath and remained there for 1 day. For solvent-exchange process, membranes were immersed into methanol for a day and after drying process, they are ready to be used.

Results and discussion

The real compositions of the dope solutions and its viscosities were tabulated as in Table1

Table 1: Multi-Components Dope Solution Formulation

Dopes formulation (wt/wt %)					
Samples	1	2	3	4	5
PES	17.26	18.91	20.42	22.71	25.26
NMP	72.21	71.03	70.06	68.74	67.38
Water	7.53	7.06	6.52	5.55	4.36
Triton x-100	3.00	3.00	3.00	3.00	3.00
Samples	1	2	3	4	5
PES	17.26	18.91	20.42	22.71	25.26
NMP	72.21	71.03	70.06	68.74	67.38

* Dope 1, 2, 3, 4 and 5 = Dope solutions

Figure 1 shows pure water permeability (PWP) of asymmetric membrane at different polymer concentration and operating pressure. , the membrane cast from PES 17.26 wt% showed the highest PWP of about 10.26 x 10⁻⁶ m³/m²s while the lowest PWP of about 6.07 x 10⁻⁶ m³/m²s was exhibited by PES 25.26 wt% membrane

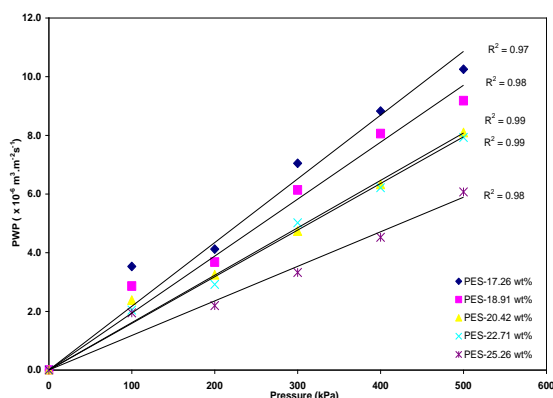


Fig.1: Pure Water Permeability (PWP) of Asymmetric NF Membranes at Different Operating Pressure and PES Concentrations.

The volume flux in Figure 2 showed that the volume flux increased with the pressure. However, it was found that the fabricated membranes performed at two different volume flux profiles. At lower polymer concentration of PES 17.26 wt% and PES 18.91 wt% the membranes exhibited higher flux. The highest volume flux of about 4.07 x 10⁻⁶ m³/m²s was

performed by PES 18.91 wt% membrane and the lowest volume flux of about 2.45 x 10⁻⁶ m³/m²s was performed by PES 25.26 wt% membranes.

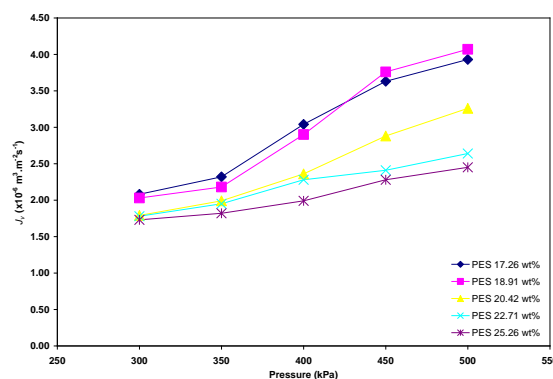


Fig.2: Volume Flux, Jv Of 0.01M NaCl at Different Operating Pressure and PES Concentrations.

Figure 3 shows that the rejection of NaCl increased with the increase of pressure. At high polymer concentration (> 20 wt%) the membranes showed a higher salt rejection. At 500 kPa, the highest salt rejection of about 45.22 % was being demonstrated by PES 25.26 wt% membrane while the PES 17.26 wt% membrane demonstrated of 21.39 % of salt rejection.

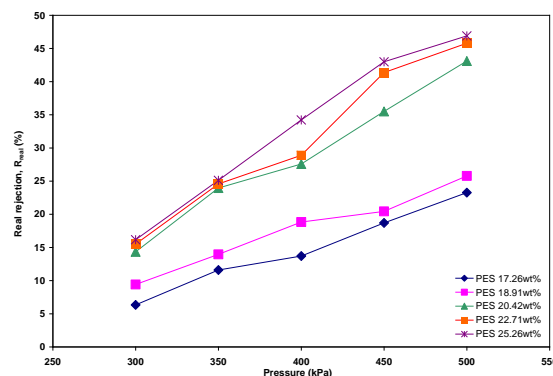


Fig.3: Real Rejection, Rreal Of 0.01M NaCl at Different Operating Pressure and PES Concentration.

In order to find the optimum polymer concentration of dope solution, the volume flux and salt rejection data at different polymer concentrations and 500 kPa of operating pressure was being plotted as in Figure 4. From the graph, it was clearly showed that there was an intersection of membranes performance close to the polymer concentration of PES 20.42 wt%. At this point, based on the performances trends, the polymer concentration of PES 20.42 wt% could be declared as the optimum polymer concentration. This finding is found to be similar as reported by the previous researchers [14,15,18].

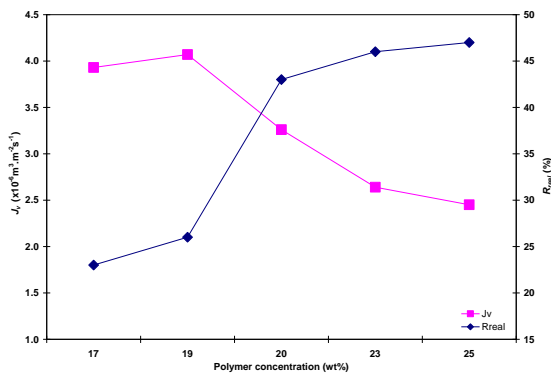


Fig.4: Effect Of PES Concentration on Jv and Rreal Of 0.01M Nacl at 500 kPa of Operating Pressure.

In Figure 29, the rejection data of difference types of multivalent salt that are Na₂SO₄, MgSO₄, MgCl₂ and NaCl at 500 kPa of operating pressure is plotted. Generally, the increasing of polymer concentration affected the rejection performance. In a sequence of rejection performance of MgSO₄> Na₂SO₄> MgCl₂> NaCl, high polymer concentration (> 20 wt %) exhibited high salt rejection of more than 80%.

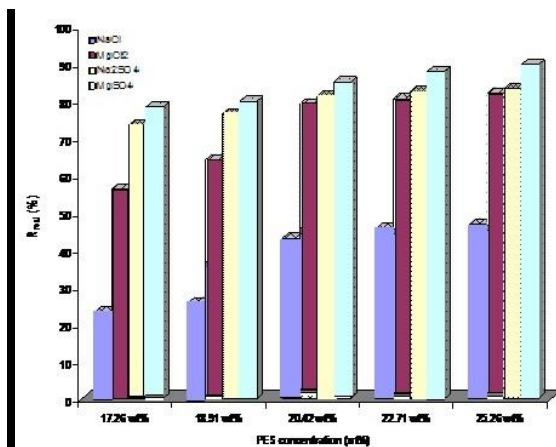
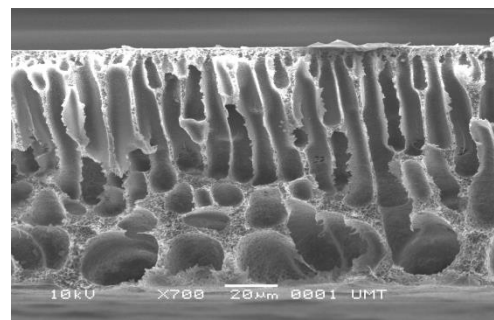
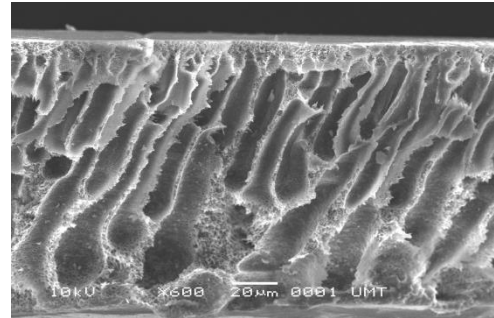


Fig. 5: Effects of PES Concentration on 0.01M Salt Rejection at 500 kPa of Operating Pressure.

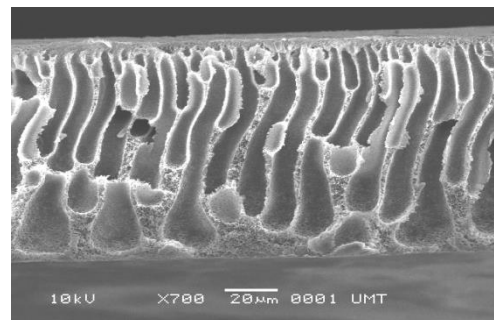
Figure 5 shows the images of cross-section morphological structures of integrally skinned-oriented asymmetric porous charged nanofiltration membranes at different PES concentrations. Nowadays, membranes morphological structure was found to be the important characteristic identifying the membrane separation ability. Such results also were reported by other researchers [16,17,19]. By using the Scanning Electron Microscopy (SEM) technique. However, at high polymer concentrations (> PES 20.42 wt%), a shorter finger like pores and spongy structure was formed beneath the skin and sub-layer. It was found that an elimination of macrovoid and minimum distribution of finger likes pore caused the reduction of volume flux and higher salt rejection.



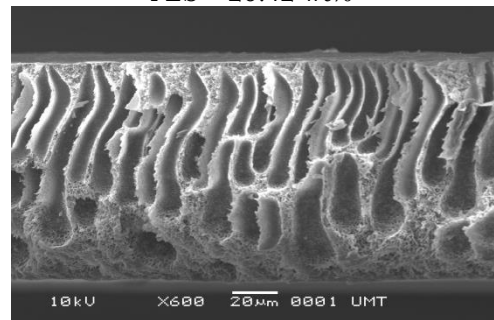
PES=17.26 wt%



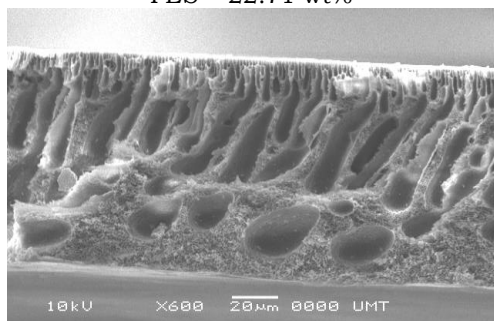
PES = 18.91 wt%



PES = 20.42 wt%



PES = 22.71 wt%



PES = 25.26 wt%

Fig.6: Cross Sectional Morphological Structures of Fabricated Asymmetric NF Membranes at Different PES Concentrations

Conclusions

The assessment on the performances of the fabricated NFS membranes provided a better understanding on the effect of PES different concentration for the fabrication of NFS membranes. Based on this study. In order to verify whether the optimum polymer concentration was PES 20.42 wt%, the pore size distributions were compared to the membranes key properties.

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