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Preparation and Characterization of Micropours Membranes Based on Modified Polyethersulfon

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Micropours polymeric membranes were prepared from homogeneous solution of Polysulfone (PSF) by phase inversion method. *N*methyl-2-pyrrolidone (NMP) was used as solvent. Polyethylene glycol (PEG) (average molecular weight 6000) was used as the polymeric additive in different concentration in the casting solution. The morphology and structure of the resulting membranes were observed by scanning electron microscope (SEM).The permeation performances of the membranes were evaluated in terms of equilibrium water content (EWC) and Porosity. Thermal and mechanical properties of membranes were analyzed by differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA) and a universal mechanical testing machine.Results showed that with increase in concentration of PEG, the pore number as well as average pore size in membranes increases. Membrane with PEG of (5wt. %) has higher (EWC) and porosity due to high porosity.

Keywords: PSF membrane, Porosity, Polyethylene glycol (PEG), Phase inversion method.

Membrane is defined as a selective barrier between two phases. The membrane has the ability to transport one component more readily than other because of differences in physical and-or chemical properties between the membrane and the permeating components[(1-5)]. Every part of a chemical process involves at least one separation or purification stage to facilitate removal and recovery of the required products[(6)]. Membrane processes are increasingly used for removal of bacteria, microorganisms, particulates, and natural organic material, which can impart color, tastes, and odors to water and react with disinfectants to form disinfection byproducts. Separations with synthetic membranes have become increasingly important. Today membrane processes are in a wide range of application and their numbers will certainly increase[(1)]. The main advantages of membrane technology are related to this unique separation principle, *i.e.* the transport selectivity of the

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membrane. Separations with membranes do not require additives, and they can be performed isothermally at low temperatures and compared to other thermal separation processes at low energy consumption. Also, up scaling and downscaling of membrane processes as well as their integration into other separation or reaction processes are easy [2,6-8]. Membranes can be classified on the bases of structure and separation principles to: porous membranes (microfiltration, ultrafiltration), nonporous membranes (gas separation, pervaporation and dialysis) and carrier membrane. Porous membranes induce separation by discriminating between particle sizes. High selectivities can be obtained when the solute size or particle size is large relative to the pore size in the membrane[7].All kinds of different synthetic materials can be used for preparing membranes. The material can either be inorganic such as a ceramic, glass, metal or organic including all kinds of polymers[1,9]. Different techniques are available to prepare synthetic membrane. The most important technique is phase inversion techniques. This technique allowing all kind of morphologies to be obtained [1,10-12]. Phase inversion is a process whereby a polymer is transformed in a controlled manner from aliquid to a solid state. By controlling the initial stage of phase transition the membrane morphology can be controlled *i.e.* porous as well as nonporous membranes can be prepared[1,13,14]. Phase inversion covers arrange of different techniques such as: solvent evaporation, precipitation by controlled evaporation, thermal precipitation, precipitation from the vapour phase and immersion precipitation. Most of the phase inversion membranes are prepared by immersion precipitation[1,11,15,-18]. Structure and properties of membranes prepared by phase inversion method depend upon many factors. Addition of additive into the casting solution is one of the major factors. An additive is used in the casting solution in order to have an optimal membrane structure. The additive can be a single component or a mixture. Generally the additive being a weak non-solventfor the polymer reduces the solvent power in the solution. A number of researchers [19-22]have reported their observation on the role of additives in the membrane structures.Eitherporous or nonporous can be obtained using the same additive, but with a variation of additive concentration or additive molecular weight. Jimenez *et al.* [21] studied the effect of Polyvinylpyrrolidone (PVP) as an additive on PES membranes and showed that the addition of PVP increased theporosity and pure water permeation of the PES membranes. Han and Nam[23] reported the effect of PVP introduction on the thermodynamic and rheological properties in PSF casting solution. The effects of PEG concentrations on the porosity of polycarbonate (PC) membranes prepared via dry/wet-phase inversion methods was studied by Deniz[24]. Other researchers studied the effect of different molecular weight PVP such as PVP K10,PVP K30, PVP K90 and PVP K360 on performance of PES membranes[25-27]. Kim and Lee[28]. investigated the effect of various molecular weights of PEG on the formation of polyetherimide (PEI) asymmetric membrane and they reported that small molecular weights of PEG such as PEG 200 and PEG 400 work as pore reducing agent for PEI membranes. PEG with molecular weight of 600, 2000, 6000 and 12,000 was used as additives to control the thermodynamics and kinetics in casting solution of PSF membranes in Kim and Lee's work[29]. Shieh *et al.* [30] reported that PEG being hydrophilic in nature, is

used to improve membrane selectivity as well as a pore forming agent. Idris *et al.* [31] found that presence of PEG of different molecular weights exhibit significant effect on performance of PES membranes. From the above literatures, although it appears that a number of works has been reported using PEG as additives, there is yet no report regarding the effect of the change concentration of PEG 6000 on morphology and performance of PSF membranes. In view of this, an attempt is made to investigate the effect of adding PEG6000 in different concentration as additives into PSF membranes. In the present work, the variations of the morphology and the structure of the PSF prepared by diffusion induced phase separation process are reported. PEG6000 of twodifferent concentration (4wt. % and5wt. %) were used as additives, separately. NMP solventwas used to prepare PSF membrane. Effects of concentration of additive (PEG) on morphology and the permeation characteristics of the prepared membrane were investigated in detail.

Experimental

Materials

Polysulfone (PSF) (average molecular weight 30,000) supplied by Sigma-Aldrich Co., USA, was used as the base polymer in the membrane casting solution. Reagent grade N-Methyl-2-pyrrolidone (NMP) (99.5% purity) was used as solvent.Reagent grade Polyethylene glycol (PEG) (average molecular weight 6000)was used as the non-solvent; pore forming; additives in the casting solution. Deionized water was used as the main non-solvent in the coagulation bath.

Membrane preparation

Micro pours PSF membranes will be prepared by phase inversion method as seen in Fig. 1. The effect of additive (PEG 6000) in two concentrations (4wt. % and 5wt. %) on membrane morphology and permeation performance are studied. The polymer (PSF) concentration was kept constant at 12wt. %, keeping the solvent and additive concentration at 88wt. %.

Without using additive

A measured amount of polymer (PSF) was dissolved in NMP solvent, at room temperature. The polymer solution was stirred for about 6 hr at room temperature using a magnetic stirrer. When the solution (consisting of polymer and solvent) became homogeneous, it was kept at room temperature for 24 hr. Subsequently, the solution was spread uniformly on a glass plate. The resulting film was then exposed for about 30 seconds to ambient temperature before immersion into coagulation bath containing water at room temperature. The casted films changed their color from transparent to white immediately after immersion into the coagulation bath and separates out of the glass plate after sometime. The prepared membrane sheets were washed under running water to remove the additional amount of additive and then kept overnight in a deionized water bath. Finally, the sheet was air dried at room temperature.

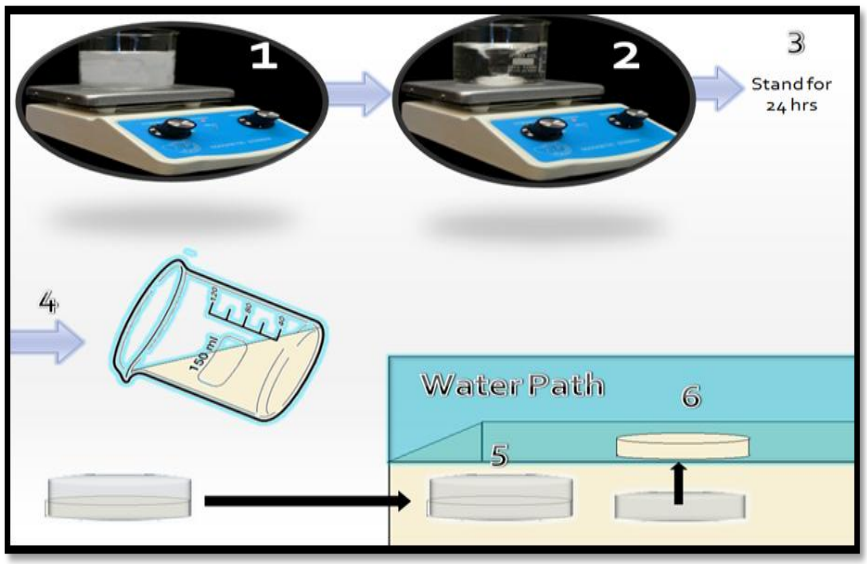

Fig. 1. Graphical representation shows membrane preparation via phase inversion method.

By using polyethylene glycol (PEG) additive

Measured amount of polymer (PSF) was dissolved in NMP solvent, at room temperature (≈25 ◦C). Solvent mixture was then mixed with the additive (PEG) to make the casting solution. The polymer solutions ware stirred for about 6 h at room temperature using a magnetic stirrer. When the solution (consisting of polymer, solvent and the additive) became homogeneous, it was kept at room temperature for 24 hr. Subsequently, the solution was spread uniformly on a glass plate. The resulting film was then exposed for about 30s to ambient temperature before immersion into coagulation bath containing water at room temperature. The casted film changed its color from transparent to white immediately after immersion into the coagulation bath and separates out of the glass plate after sometime. The prepared membrane sheet was washed under running water to remove the additional amount of additive and then kept overnight in a deionized water bath. Finally, the sheet was air dried at room temperature.

Membrane characterization

The prepared membranes were characterized by morphological and permeation performance, thermal and mechanical analysis.

Morphological analysis

The shape and size of the pores as well as pore size distribution and porosity are important parameters deciding the separation performance. The morphology of the prepared membranes was investigated by microscopic observations.

Microscopic observation: Microscopic observation was carried out by a Scanning Electron Microscope (SEM). Using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., magnification14x up to 1000000 and resolution for Gun.1n which directly provides the visual information of the top surface as well as cross-sectional morphology and porosity of the membranes. Computerized analysis of SEM image is a standard and widely used method for the investigation of perforated materials[32,33]. The membrane was coated with gold by (K550X sputter coater, England) then scanned by scanning electron microscope.

Equilibrium water content (EWC): Equilibrium water content is considered to be an important characterization parameter as it indirectly indicates the degree of hydrophobicity of a membrane[34]. Also, it is related to the porosity of a membrane. Membranes were weighed in an electronic balance in wet state after mopping the surface water with a clean tissue paper. The wet membranes were dried by putting in a vacuum oven for 24 hr at a temperature of 50–60 ◦C and again they were weighed in dry state. Then the equilibrium water content (EWC) at room temperature is calculated as follows:

$$\text{EWC (\%)} = \frac{Ww - Wd}{Ww} \times 100$$

Where, *W*w is weight of wet membranes (g) and *W*d is weightof dry membranes (g).

Porosity: Porosity of the membrane plays an important role on permeation and separation. The membrane porosity is determined as follows [35].

$$\text{Porosity} = \frac{Ww - Wd}{\rho w \times V}$$

Where, ρw is density (kg/cm^3) of pure water at room temperature and *V* is volume of the membrane in wet state (m^3).

Thermal characteristics

Differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA) data are valuable for studying the thermal propertiesof micro porous polymeric membranes. Simultaneous DSC-TGA model SDT Q600 (USA) was used in the temperature range of 25 to 600 ^{0}C. A heating rate of 10 ^{0}C / min was used and the purge gas used was nitrogen.

The mechanical properties

The tensile strength of membranes was measured with a universal mechanical testing machine, SHIMADZUCORPORATION (JAPAN), model (UH-2000KNA,NO121203500007), capacity (2000, 1000,400,200,100,40)KN and working conditions (voltage ±10% ,warm. up 15min,temperature 5- 40^{o}C).

Results and Discussion

Morphological characteristic

Microscopic observation

Microscopic study through SEM analysis was carried out for the prepared membranes to give qualitative information about their surface morphology. SEM image of the surface of membrane prepared without using the additive has the smallest pores with diameter of less than 4µm (Fig. 2). Meanwhile the largest pore diameter of 11µm and with asymmetric structure with homogenous distribution of pores was obtained for membrane prepared by using 5 Wt. % of PEG (Fig. 4).An inter medium pore diameter 8 to 5 µm was achieved for membrane prepared by using 4 Wt. % PEG (Fig. 3).It can be concluded that the increase in PEG concentration increases the pore numbers and their average pore diameter.

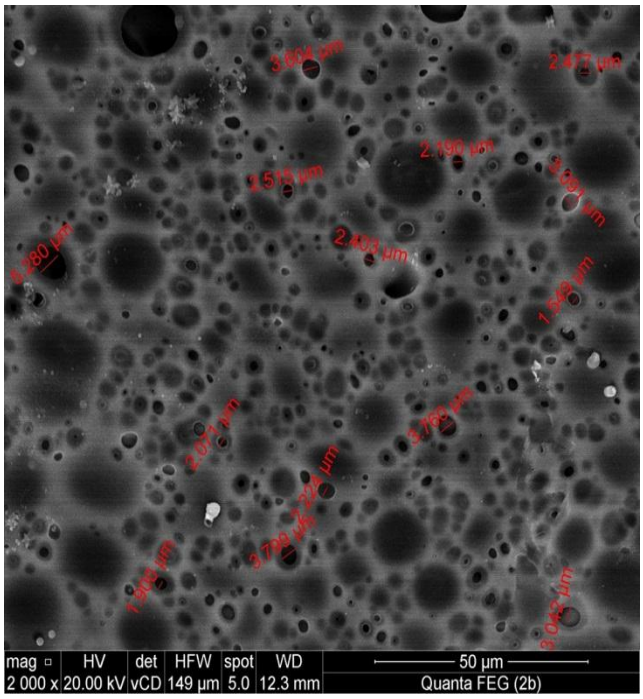

Fig. 2. SEM image of the prepared membrane without using additive.

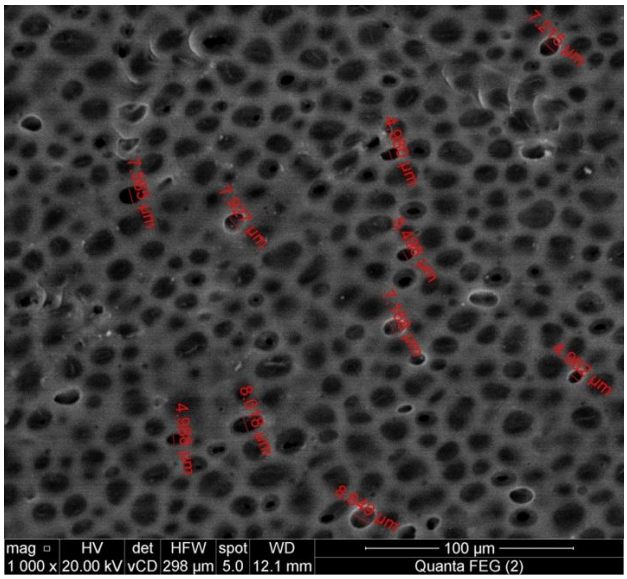

Fig. 3. SEM image of the prepared membrane using 4 wt% of PE .

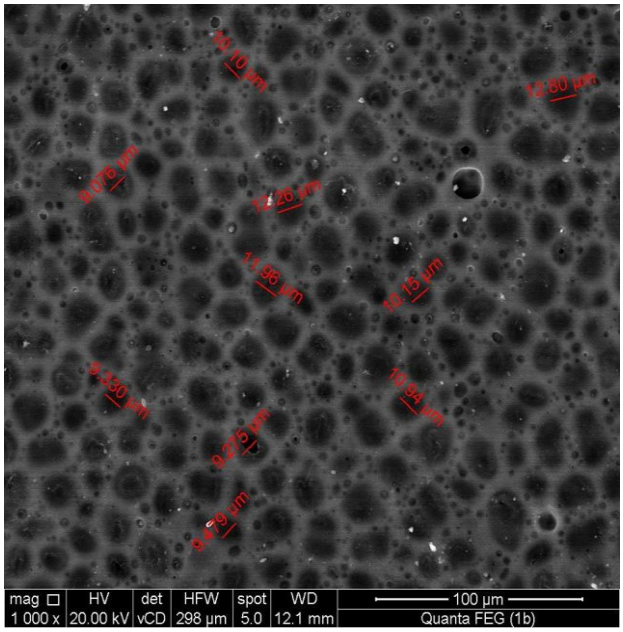

Fig. 4. SEM image of the prepared membrane using5 wt% of PEG .

Equilibrium water content (EWC)

The equilibrium water content is an important parameter for membrane characterization. The EWC of different membranes is presented in Table 1. It can be noted that, the membrane prepared without using PEG additives gives the lowest EWC (25 Wt. %). Meanwhile the addition of 4 &5 Wt. % of PEG additive during the preparation of membrane elevates its EWC to 39 and 41 Wt. % respectively. This increase in EWC confirms the presence of increasing number of pores in the membrane with the presence of additives (PEG) (as discussed in Section 3.1). The pores on the surface as well as cavities in the sublayer are responsible for accommodating water molecules in the membranes[36].

Porosity

Porosity of membrane is calculated.Data are presented in Table 1. It can be observed that the porosity of the membranes prepared in the presence of 4 and 5 Wt. % of PEG additives is higher the porosity of the membrane prepared in absence of additives. The variation of porosity may be explained on the basis of thermodynamic and kinetic consideration. Addition of an additive into the casting solution has two effects. Firstly, it causes thermodynamic enhancement of the phase separation by reducing the miscibility of the casting solution with the nonsolvent; this results in instantaneous demixing. Secondly, it causes kinetic hindrance against phase separation by increasing the viscosity of the solution; this results in delayed demixing[37-39]. Increase in viscosity increases the ratio of non-solvent inflow to solvent outflow which according to the theory suggested by Young *et al.* [40] results in a more porous membrane. Thus, the increase in porosity in membranes with PEG 6000 may be due to decrease in miscibility of the casting solution with water with addition of PEG 6000. This in turn, can work in favor of the thermodynamic enhancement in the demixing of casting solution.

TABLE 1. Separation performance of membranes .

Characteristics	Without additives	Percent of additives (Wt %)	
		4%	5%
Water content	25	39	41
Porosity	0.06	0.09	0.1

Thermal characteristics

Thermal properties of prepared membranes were determined using DSC and TGA. The analysis of the changes in stability and thermal properties of membranes is important for their application, as well as the characterization and determination of their chemical and physical changes. The prepared membranes using DSC-TGA instrument was determined as shown in Fig. 5-7. It can be noticed from the figures that the thermal stability of membranes prepared by

using PEG additive gives higher value; up to 450°C and 500°C by using 4 and 5wt. % PEG additives respectively; than that prepared in the absence of PEG additive as it gives thermal stability up to 320°C.

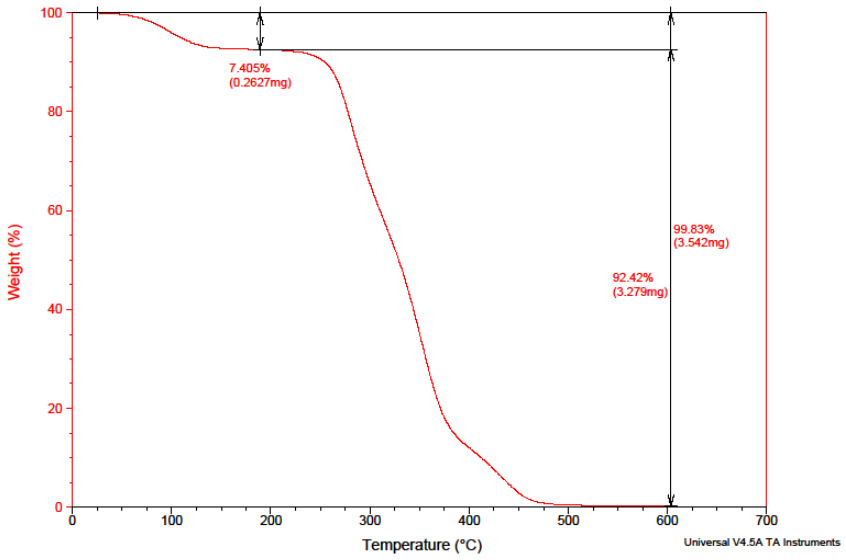

Fig. 5. DSC-TGA Curve of the prepared membrane without using PEG6000 .

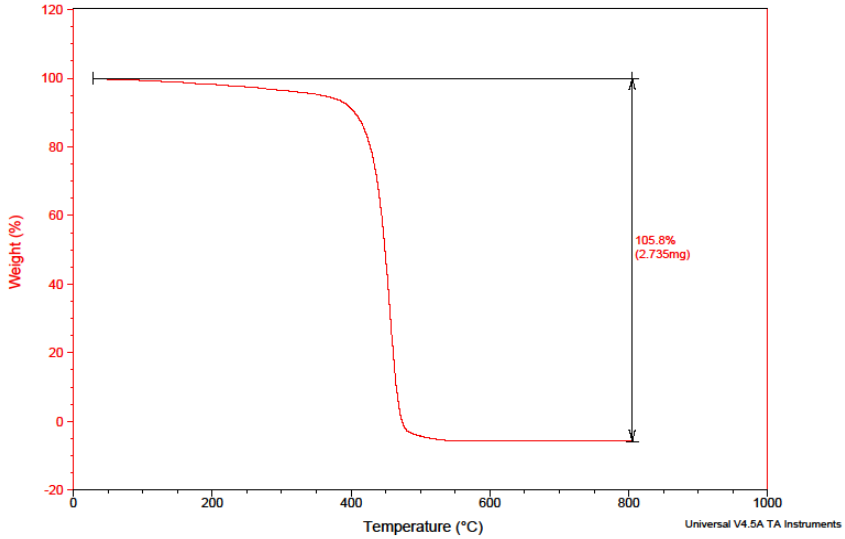

Fig. 6. DSC-TGA Curveof the prepared membrane using 4 wt% of PEG additive .

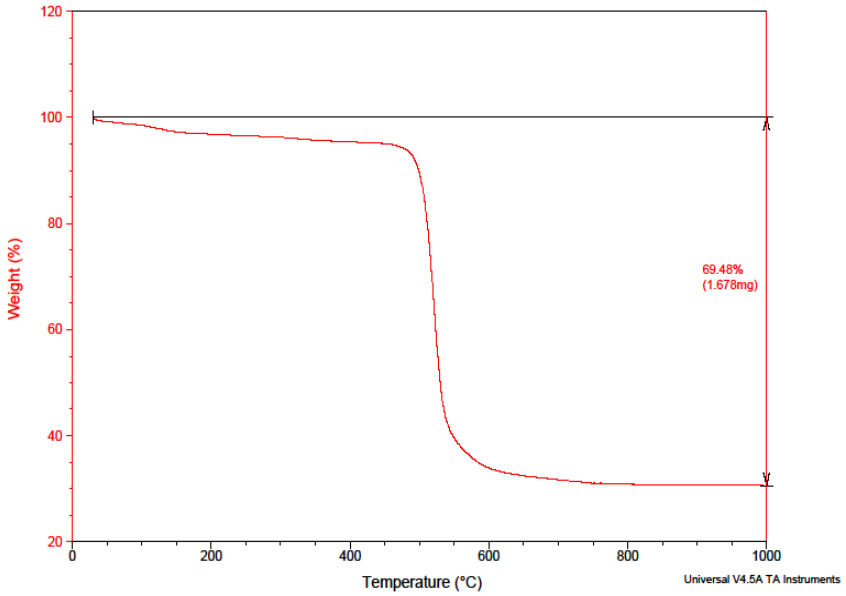

Fig. 7. DSC-TGA Curve of the prepared membrane using 5wt% of PEG6000.

Mechanical characteristic

The tensile strength and elongation at break for prepared membranes were analyzed. This test determines the main mechanical characteristics of the material and thus indicates its mechanical durability and its plausible application. Data are presented in Table 2.It can be noticed that the increase of PEG additive during membrane preparation increases its mechanical stability. As the membrane prepared without using PEG as additive gives the lowest values in strength at break and elongation percentage of 9.1168 MPa and 13.8480 MPa respectively. Meanwhile, the membrane prepared by addition of 4 and 5 Wt. % of PEG additive chows higher values in its tensile strength reaches to 12.1557 and 12.4756 MPa respectively and its elongation percent reaches to 15.1440 and 18.0160 respectively.

TABLE 2. Mechanical Properties of the prepared membranes.

Samples	Tensile N/mm^2 MPa	Elongation %
PSF Alone	9.1168	13.8480
PSF + PEG (4%)	12.1557	15.1440
PSF + PEG (5%)	12.4756	18.0160

Conclusion

Micropours PSF membranes were prepared from casting solutions containing12 wt. % of PSF with NMP solvent, using diffusion induced phase separation process. PEG of average molecular 6000 was used as additive. Effects of different concentrations of PEG on the morphology, thermal and mechanical properties of membranes were studied in detail. The permeation performance of the prepared membranes with different concentrations of PEG was also evaluated in terms of EWC and porosity. The observations can be summarized as follows:

- Allthemembraneswerefoundtohaveasymmetricstructureasseenfrom SEMphotographs.
- WithincreaseinconcentrationofPEG,theporenumber aswellastheaverageporeofthe prepared membranewasincreased.
- TheEWCandporositywereseentoenhance greatlywithincreaseinconcentrationofPEG.
- Alsothe thermal and mechanical stability for 5wt.% of PEG is higher than 4wt.% of PEG. This means that the increase in concentration of PEG6000 increases thermal and mechanical stability increase.

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تحضير وتوصيف اغشيه فى حجم الميكرو معتمده على البولى ايثر سلفون المعدل

ريـم كمال كامل فـرج ، نيرمين حفنى محمـد* ، سمـاح عبد الهادى محـمدابوالفتوح* ، امال سعيد فرج * ، فتحى سمير* ، شيماء محمد السعيد و رباب محمـد ابوشهبه**

قسم الاستخدامات ، *قسم التكرير - معهد بحوث البترول و **قسم الكيمياء - كلية العلوم - جامعة الازهر- القاهرة - مصر

تم تحضر اغشيه بوليمريه فى حجم الميكرو بواسطه البولى ايثر سلفون المذابه فى الميثيل بيريليدون كمذيب بطريقة انعكاس الحاله. تم استخدام تركيزين من البولى ايثيلين جليكول كماده محبه للماء لتحسين خواص مسامية غشاء البولى ايثر سلفون. تم دراسة الخواص السطحيه وتعيين حجم مسامية الاغشيه المحضرة. تم تقييم كفاءة الاغشيه المحضرة بتعيينتوازن محتوى الماءوالمسامية. تم دراسة الثبات الحرارى والخواص الميكانيكيه للبوليمرت المحضرة. اثبتت النتائج ان حجم مسامية الغشاء وتوازن محتوى الماء تزيد بزيادة تركيز البولى ايثيلين جليكول.