



Concentration of gelatin solution with polyethersulfone ultrafiltration membranes

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ABSTRACT

In this work, polyethersulfone (PES) flat sheet ultrafiltration (UF) membranes were prepared by immersion precipitation phase inversion process with polyvinylpyrrolidone (PVP 30K) and acetone as additives. The best preparation condition for PES membranes with high water flux and rejection (to BSA) was decided. It was found that the optimal composition of the polymer casting solution was: 16 wt% (PES), 2 wt% (PVP 30K), and 1 wt% (acetone). Pure water flux of the membrane prepared at this condition reached to 373 L/m² h at 0.1 MPa, and the rejection to BSA was 91%. Compared with other reports, the rejection was slightly low but the flux of the PES membrane was high. When the membrane was used to concentrate gelatin solutions, the rejection value was over 75%. It was found that increasing the feed temperature and transmembrane pressure enhanced the permeation flux, but the rejection decreased slightly. However, increasing the cross-flow velocity of the feed solution simultaneously increased both the permeation flux and the rejection.

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Keywords: Gelatin; Polyethersulfone; Polyvinylpyrrolidone; Ultrafiltration

1. Introduction

Gelatin is a water soluble protein (20–250 kDa) prepared from animal skin and bones. Gelatin is composed of a unique sequence of amino acids. Fig. 1 shows the chemical structure of gelatin which contains the repeating sequences of glycine–X–Y triplets, where X and Y are frequently proline and hydroxyproline amino acids (Structure and aminoacid composition, 2010). Traditionally, gelatin is applied to three areas: food, pharmaceutical, and photographic industries. In the food industry, gelatin is often added in desserts and confectionery. In the pharmaceutical industry, gelatin is used to make the shell of capsules. In the photographic industry, gelatin is a necessary component for the preparation of silver halide emulsions in the production of photographic film (Karim and Bhat, 2008). In recent years, gelatin was adopted as the binder for sulfur cathode of rechargeable lithium–sulfur batteries (Sun et al., 2008).

Pigskin is the most significant raw material for the production of gelatin (Karim and Bhat, 2008). Fig. 2 shows a representative process of producing gelatin. First, pigskin is washed with cold water and soaked in hydrochloric acid or

sulfuric acid for hydrolysis. Then, the pigskin is extracted by successive applications of hot water at gradually increasing temperature to minimize thermal degradation. The first extraction is conventionally operated at 50–60 °C. The temperature is increased by 5–10 °C until the final extraction is carried out close to the boiling point. The pH value, time, temperature, and number of extractions vary from manufacturer to manufacturer and depend on the product needs, type of equipment and economics. After the extraction stage, the extracted dilute gelatin solutions are filtered, concentrated, and deionized. The conventional method of concentration is by evaporators in three stages: preliminary concentration, main concentration, and finishing stage for high concentration. The conventional method has some shortcomings, such as consuming lots of energy, emitting hydrocarbons to atmosphere when oil is used to heat the evaporators, and degrading the protein molecules of gelatin (Dutr  and Tr g rdh, 1995; Simon et al., 2002).

Concentration by the conventional evaporation processes is being replaced by ultrafiltration (UF) membrane technique as shown in Fig. 2. Inorganic tubular UF membranes are generally used. Compared with the conventional concentration technique, there are at least three advantages when using UF

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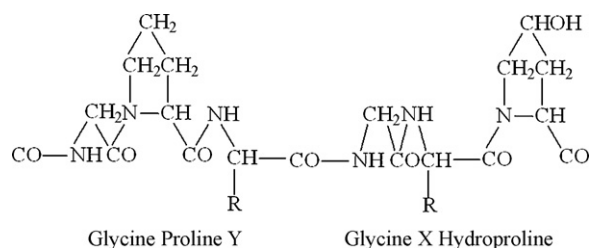


Fig. 1 – Chemical structure of gelatin.

membranes. The total operation cost is low although the initial purchasing cost for UF membranes is high. The product quality is improved because less degradation occurs, particularly for the initial concentration stage, and the emissions of hydrocarbons can be reduced. After the concentration stage, the gelatin solution is cooled and extruded as noodles. The noodles are deposited onto a stainless steel belt and dried with air. Finally, the dried gelatin is broken into powder or pellets (Dutr  and Tr g rdh, 1995; Simon et al., 2002).

Polymeric UF membranes are another type of separation membrane which have been widely used to separate micro-solutes from water solutions in the pharmaceutical, biotechnology, medical, food, beverages, potable water, dairy, metallurgical and electro-coating industries (Ulbricht, 2006). Compared with inorganic membranes, the product cost of polymeric membranes is low. To our best knowledge, however, there is only one paper on concentrating gelatin solution with polymeric UF membranes by Dutr  and Tr g rdh (1995) except this work. The possible reason is that the permeation flux is not large enough when concentrating gelatin solution.

Polyethersulfone (PES) is an important high performance material. It was firstly manufactured by ICI Company in 1972. PES possesses many excellent merits, such as high mechanical strength, thermal stability and chemical resistance. In addition, it has low creep properties, flame retardancies, excellent insulation properties and high dielectric strength. Therefore, PES is widely used in aviation, microelectronics, automobile, and membrane separations (Product Brochure, 2010). For membrane separations, PES has been prepared into ultrafiltration, nanofiltration and reverse osmosis membranes. Furthermore, it is made into hemodialysis membranes due to its perfect biocompatibilities (Kim and Kim, 2005; Zhang and Shi, 2009; Ulbricht, 2006). To enhance the performances of PES UF membranes, some additives are often added to polymer casting solution, such as polyvinylpyrrolidone (PVP) or polyethylene glycol (PEG) (Ahmad et al., 2005; Barzin et al., 2004; Kim and Kim, 2005; Mosqueda-Jimenez et al., 2004; Qin et al., 2005a; Rahimpour et al., 2007; Rahimpour and Madaeni, 2007; Wang et al., 2006). Commercial UF membranes made

from PES are mostly produced by using PVP as an additive (Ulbricht, 2006).

In this paper, two research works are reported. First, PES flat sheet UF membranes were prepared using the phase inversion technique with PVP (30K) and acetone as a mixed additive. The pure water flux (PWF), rejection to bovine serum albumin (BSA), and membrane morphology were characterized. Second, the prepared membranes were used to concentrate gelatin solutions. The effects of operation conditions on the concentration performances of the membranes were researched in detail.

2. Materials and methods

2.1. Materials

PES (Ultrason E 6020P) was purchased from BASF Co., Germany. PVP (30K) was purchased from Fluka Chemika, Switzerland. Acetone and N-methyl-2-pyrrolidone (NMP) were purchased from Tianjin Kermel Chemical Reagents Development Centre, China. Bovine serum albumin (BSA) (68,000 Da) was purchased from Beijing AoBoXing Bio-tech Co., Ltd., China. Gelatin was purchased from Hengshui Jingyu Gelatin Ltd., China. Distilled water was used throughout this study.

2.2. Preparation of UF membranes

PES ultrafiltration membranes were prepared by immersion precipitation phase inversion method. PES was dried at 100 °C for at least 24 h before use. Polymer casting solutions were firstly prepared by mixing PES and PVP with the mixture of NMP and acetone at room temperature. Then, homogeneous casting solutions were formed after the above mixtures had been maintained at 50 °C for 4 h. According to the published papers on preparing PES UF membranes (Ahmad et al., 2005; Barzin et al., 2004; Kim et al., 2005; Kim and Kim, 2005; Qin et al., 2005b; Rahimpour et al., 2007; Rahimpour and Madaeni, 2007; Wang et al., 2006), the concentrations of PES in casting solutions were generally 15–18 wt%. In this experiment, the concentration of PES in the casting solution was maintained at 16 wt%. First, the casting solutions were cast on non-woven fabric supports (MF110, Vilene, Japan) at room temperature. After 40 s exposure to the air, the nascent membranes were immersed in a water bath maintained at 20 °C and kept in the water bath at room temperature for at least 48 h until all of solvent was removed.

2.3. Ultrafiltration experiment

The ultrafiltration experiment was carried out on a self-made equipment. Fig. 3 shows the schematic diagram of the experimental setup. Feed solutions or water were loaded in the feed tank. They were driven from the feed tank into the membrane cell under the pressure of compressed nitrogen gas. The pressure on the feed side was maintained constantly by the nitrogen gas. The permeation was immediately transported in a collecting flask by a peristaltic pump (Pump 2). When the rotating speed of the peristaltic pump was regulated to a suitable speed, there was no pressure on the permeate side. Therefore, the transmembrane pressure equalled to the pressure on the feed side. After flowing across the membrane cell, the feed solution entered in a sealed buffer tank and was returned in the feed tank with another peristaltic pump (Pump 1), which had no influence on the pressure of the feed side.

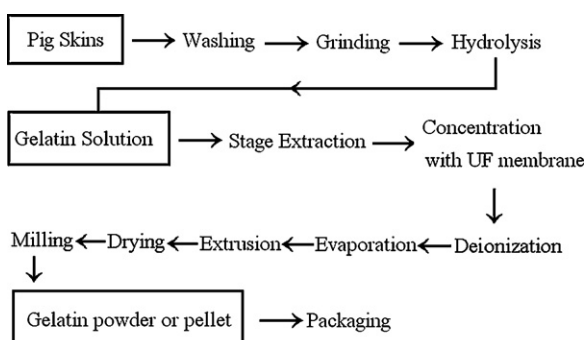


Fig. 2 – Gelatin production process.

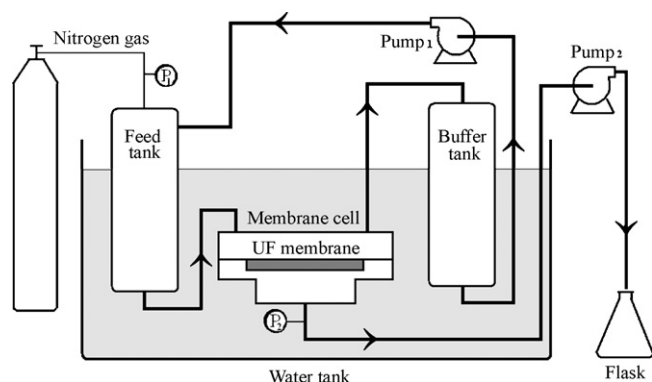


Fig. 3 – Schematic diagram of the experimental setup of UF membrane for the concentration of gelatin solution.

The cross-flow velocities of the feed solutions could be regulated through controlling the rotating speed of Pump 1. When researching the influence of cross-flow velocity of gelatin solution on the permeation performances, the cross-flow velocity on the feed side was regulated between 0.06 and 1.20 cm/s with the pump.

Three membranes cut from one prepared large membrane were used at each measuring condition. For each membrane sample, firstly, the transmembrane pressure was controlled at 0.20 MPa for 30 min with water as the feed to pre-press the membrane to reaching a stable state. Then, water was changed with feed solution. After circulating the feed solution for 5 min, all membrane samples were tested under the transmembrane pressure of 0.10 MPa at room temperature. Permeation flux or pure water flux and rejection to BSA were measured. The permeation flux, J (L/m² h) was calculated by the following equation:

$$J = \frac{V}{At} \quad (1)$$

where V was the volume of permeation, A was the membrane area (7.0 cm²), and t was the permeation time. The rejection, R (%) was calculated by the following equation:

$$R = \left(1 - \frac{C_p}{C_f} \right) \quad (2)$$

where C_p and C_f (mg/mL) were the concentrations of permeation and feed solution, respectively. For the BSA rejection test, the concentration of BSA in phosphate-buffered solution (composed of NaH₂PO₄ and Na₂HPO₄, pH = 7.4) was 1.0 mg/mL. For the experiment of concentrating gelatin solutions, the concentration of gelatin feed solutions was 1–7 wt%. The concentrations of BSA and gelatin in the permeation and feed side were measured at 280 nm by a UV-vis spectrophotometer (T6, Beijing Purkinje General Instrument, China).

2.4. Characterization of membrane morphology

The structure of the prepared membranes was examined by a scanning electron microscope (FEI Quanta 200, USA). The samples were frozen in liquid nitrogen and fractured. After sputtering with gold, the cross-section of the membranes was viewed with the SEM.

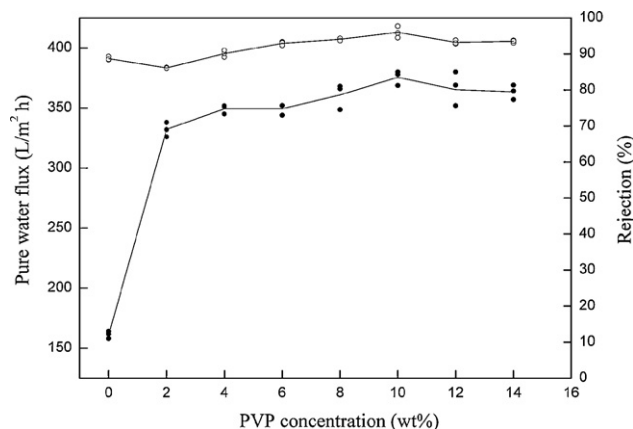


Fig. 4 – Effect of PVP concentration on PWF and rejection.

3. Results and discussion

3.1. Effects of preparation conditions on performances of PES membranes

3.1.1. Effect of PVP concentration on performance of membranes

The effect of PVP concentration (0–14 wt%) in the casting solutions on the performances of PES membranes is shown in Fig. 4. It shows that with the increase of PVP concentration (0–10 wt%) in the casting solutions, PWF (pure water flux) firstly increases and reaches to a maximum value (375 L/m² h) when the PVP concentration is about 10 wt%. When the PVP concentration is only 2 wt%, the PWF reached 330 L/m² h, which is only slightly lower than the maximum value but is twice the PWF (160 L/m² h) of the membrane without adding PVP. It means that adding small amount of PVP can significantly enhance the PWF. The rejection to BSA increases slowly with the increase of PVP concentration. The effect of adding PVP on the formation and performances of membranes had been widely studied (Jin et al., 2007; Jung et al., 2004, 2005). The common viewpoint was that PVP was a kind of hydrophilic additive which could be dissolved out by water. After the nascent polymer membrane was immersed in water, the original sites occupied by PVP would become micropores, and the pore number would increase when more PVP was added in casting solution (Kim and Kim, 2005). Generally, most of PVP could be dissolved out by water, but small quantity of PVP would remain in the final membrane. Consequently, when more PVP was added, more PVP would remain in the membranes which induced the flux to be constant or slightly to decrease.

3.1.2. Effect of acetone concentration on performance of membranes

Acetone is an effective additive which has been often used by researchers to regulate membrane structure in the immersion precipitation phase inversion technique (Barth et al., 2000; Wang et al., 2002). We found that when acetone was added in the casting solutions (2 wt% PVP was added), the performance of the membranes could be further enhanced as shown in Fig. 5. The acetone concentration was changed from 0 to 11 wt%. It shows that with the increase of acetone in the casting solutions, PWF and rejection all increase firstly then decrease. When the concentration of acetone is 7 wt%, PWF reaches the maximum value, 401 L/m² h, and the corresponding rejection to BSA is 94%. Acetone is a volatile

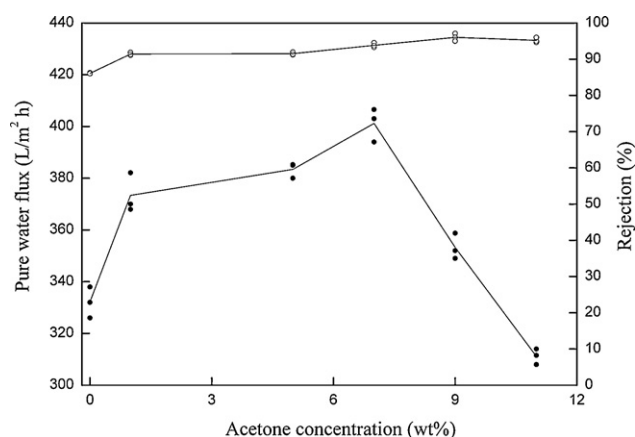


Fig. 5 – Effect of acetone concentration on PWF and rejection (PVP concentration in casting solutions is 2 wt%).

organic solvent. When acetone evaporated, the top separation lay of the membranes became thin which induced the flux to increase. However, when more acetone was added, the polymer concentration in the top lay of the nascent membranes would increase after acetone evaporated. After the nascent membranes were immersed in water, the size of the formed porous would become small which induced the flux to decrease (Wang et al., 2002).

3.1.3. Morphology of membranes

Fig. 6 shows the morphology of cross-section of the PES membrane which was prepared at the condition that the concentrations of acetone and PVP were 1%, and 2%, respectively. It is apparent that the membrane has a typical asymmetric structure which consists of a thin dense skin layer and fine porous sublayer with many finger-like cavities. According to the shown scale, the thickness of the dense layer is about 0.2 μm . Fig. 7 shows the inside morphology of a finger-like cavity of the membrane. There are many small holes in the cavities. The reason for the large permeation flux of the membranes maybe has a relation with these small holes.

3.1.4. Comparison with other PES UF membranes

Table 1 lists some preparation conditions and performances of asymmetric ultrafiltration membranes studied in recent

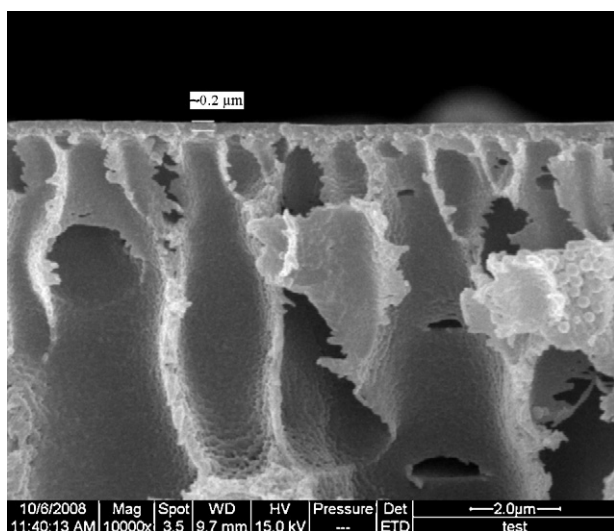


Fig. 6 – Morphology of cross-section of PES membrane at 10,000 \times .

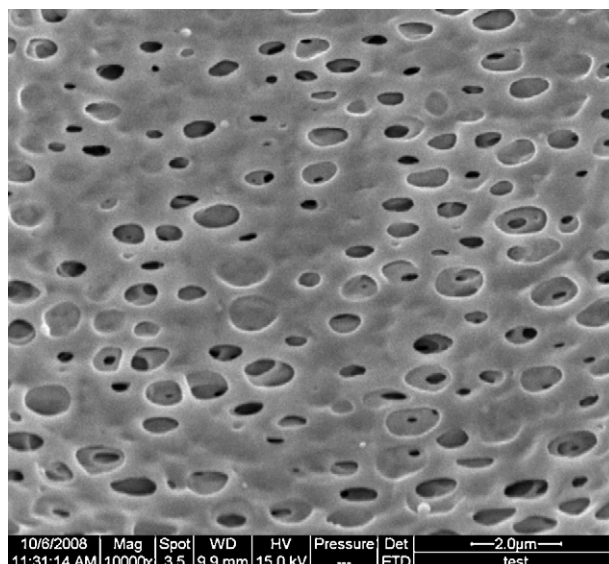


Fig. 7 – Inside morphology of cavity of PES membrane.

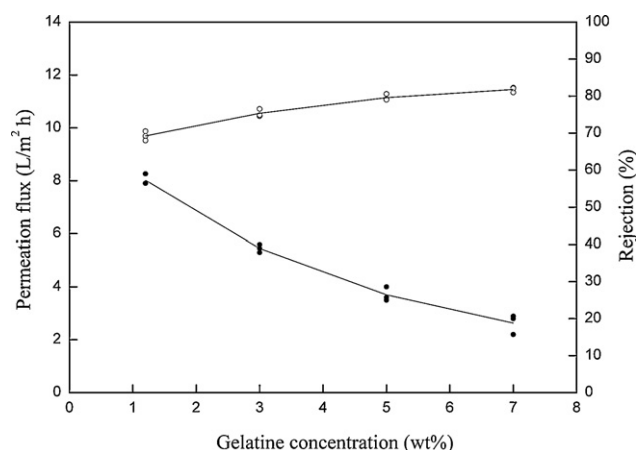


Fig. 8 – Effect of concentration of gelatin solutions on permeation flux and rejection (temperature 55 °C; pressure 0.10 MPa; cross-flow velocity of feed 0.67 cm/s). ●: permeation flux; ○: rejection.

years by adding PVP as additives. To give an easy comparison of the pure water fluxes, the original different units were consolidated into the same unit: (L/m² h Pa). Because many operating pressures were different, some flux data were also re-calculated from the original data. Compared with these flat sheet UF membranes, the rejection of the prepared PES membrane is slightly low but the flux is high.

3.2. Concentration gelatin solutions with the membrane

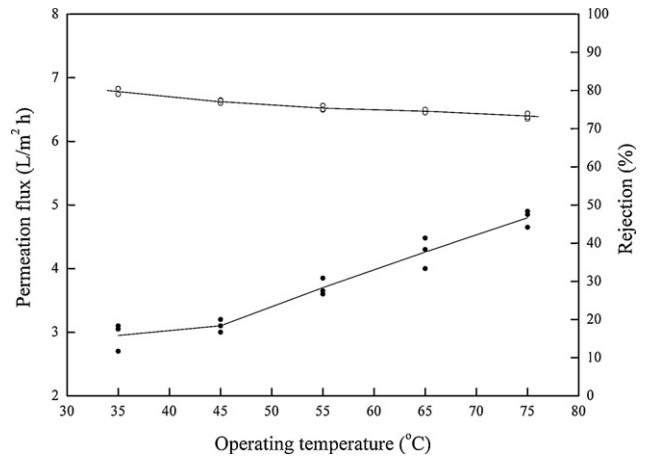
The PES membrane (concentration of acetone and PVP was 1 and 2 wt%, respectively) was used to concentrate gelatin solutions. The effects of operation conditions on the performances of the membrane were researched as follows.

3.2.1. Effect of concentration of gelatin solutions on performances of the membrane

Fig. 8 shows the effect of the concentration of gelatin solutions on the permeation flux and the rejection. It shows that with the increase of gelatin concentration, the permeation flux decreases, but the rejection slightly increases. Simon et al. (2002) found a similar phenomenon when they used Al_2O_3

Table 1 – Preparation conditions and performances of asymmetric ultrafiltration membranes.

Casting solution composition	Concentration of polymer (wt%)	Concentration of PVP (wt%)	External coagulant composition	Pure water flux ($\times 10^{-5}$ L/m ² h Pa)	Pressure (MPa)	Rejection (%)	Membrane type	Reference
PES/DMAC/1,2-PD/PVP (K90)	15	5	Water	1565	0.1	97.6 (BSA)	Hollow fiber	Qin et al. (2005b)
PES/NMP/PVP(40K)	25	9.9	Water	110	0.1	97.4 (humic acid)	Hollow fiber	Qin et al. (2005a)
PES/DMAC/PVP (K90)	18	3/6	Water	55–71	0.3	60 (Dextran 18K)	Hollow fiber	Barzin et al. (2004)
PES/DMAC/PVP(25K)	16	2	Water/2-propanol	37	0.3	98 (milk)	Flat sheet	Rahimpour and Madaeni (2007)
PES/DMAC/PVP(25K)	16	2	Water/2-propanol	57	0.3	96 (milk)	Flat sheet	Rahimpour et al. (2007)
PES/DME/PEG/PVP(60K)	18	3	Water	40	0.1	98 (BSA)	Flat sheet	Wang et al. (2006)
PSE/NMP/P(VP-AN)	17	5	Water	6	0.3	88.6 (Dextran 40K)	Flat sheet	Kim et al. (2005)
PES/NMP/P(VP-S)	17	5	Water	14	0.3	50 (PEG 10K)	Flat sheet	Kim and Kim (2005)
PSE/NMP/PVP(10K)	18	15	Water	97	0.1	20 (PEG 20K)	Flat sheet	Ahmad et al. (2005)
PES/NMP/PVP(10K)	12/18	6/18	Water	10–20	0.3	–	Flat sheet	Mosqueda-Jimenez et al. (2004)
PES/NMP/PVP(30K)/Acet(1%)	16	2	Water	373	0.1	91 (BSA)	Flat sheet	This work
PES/NMP/PVP(30K)/Acet(7%)	16	2	Water	401	0.1	94 (BSA)	Flat sheet	This work

**Fig. 9 – Effect of operating temperature on permeation flux and rejection (concentration 5 wt%; pressure 0.10 MPa; cross-flow velocity of feed 0.37 cm/s). ●: permeation flux; ○: rejection.**

membranes to concentrate a fish gelatin solution. In their experiment, they found that when the feed concentration increased, the flux nearly decreased linearly with increasing concentration, but the rejection was not affected by the feed concentration. For this experiment, we speculate the reason for the rejection slightly increasing with the feed concentration was that the available average pore size of the membrane became smaller because more gelatin molecules accumulated on the surface of the membrane when the feed concentration became larger. Compared with the pure water flux listed in Table 1, the descent of the permeation flux is rapid. The reasons maybe came from two aspects. One was concentration polarization, another was pore plugging. However, confirming the reasons needs more detailed research work that exceeds the research content of this work. The rejection values are over 70%, which mean that at least half of water in the gelatin solutions can be removed by the membrane.

3.2.2. Effect of operation temperature on performances of the membrane

Fig. 9 shows the effect of the operation temperature on the permeation flux and the rejection. It shows that the permeation flux increases when the temperature increases, but the rejection decreases slightly. When the temperature was increased, the viscosity of gelatin solutions decreased, which induced the fouling degree decreased because the molecular interactions between gelatin protein and membrane surface became weak when the temperature increased (van Oss, 1994). Consequently, the permeation flux increased. However, increasing the operating temperature above 80 °C for long time will cause degradation because gelatin is heat sensitive matter. Therefore, increasing temperature above 80 °C is not recommended in practice.

3.2.3. Effect of operation pressure on performances of the membrane

Fig. 10 shows the effect of the operation pressure on the permeation flux and the rejection. It shows that when the pressure is changed from 0.05 to 0.2 MPa, the flux almost linearly increases. However, after the pressure reaches to 0.3 MPa, the flux does not increase anymore. The reason was ultrafiltration separation was a pressure driven membrane process, increase of pressure could directly induce the permeation flux

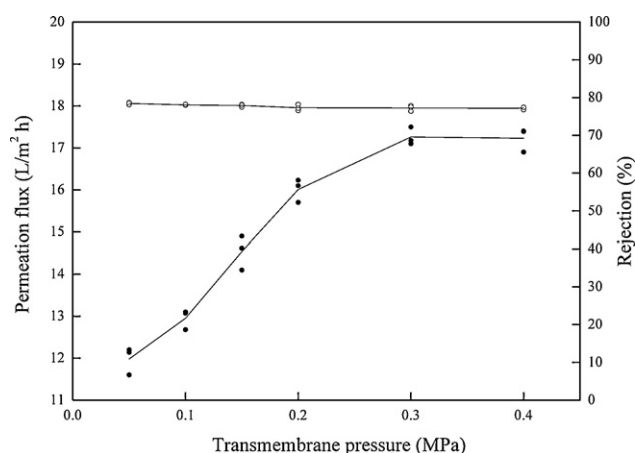


Fig. 10 – Effect of transmembrane pressure on permeation flux and rejection (concentration 1 wt%; temperature 55 °C; cross-flow velocity of feed 0.37 cm/s). ●: permeation flux; ○: rejection.

to increase. However, when higher pressure was applied to the feed side, the concentration polarization closing to the membrane surface became serious, which limited the increase of the permeation flux.

3.2.4. Effect of cross-flow velocity of feed solutions on performances of the membrane

Generally, the change of cross-flow velocity of the feed solution can distinctly influence the separation performance of UF membranes because the concentration polarization and membrane fouling from macro-molecular solute can be weakened when the cross-flow velocity of feed was increased (Choi et al., 2005). Fig. 11 shows the effect of cross-flow velocity of the feed solutions on the permeation flux and the rejection. It shows that both the permeation flux and the rejection increase with the increase of the cross-flow velocity, but the relative increase of rejection is smaller than the relative increase of flux. The reason was that when the cross-flow velocity of the feed was increased, the concentration polarization and membrane fouling from gelatin were weakened. Therefore, water flux in the permeation absolutely increased. Since the increased extent of water flux was relatively larger than the increased extent of gelatin in the permeation, the rejection value became large. Compared with increasing temperature,

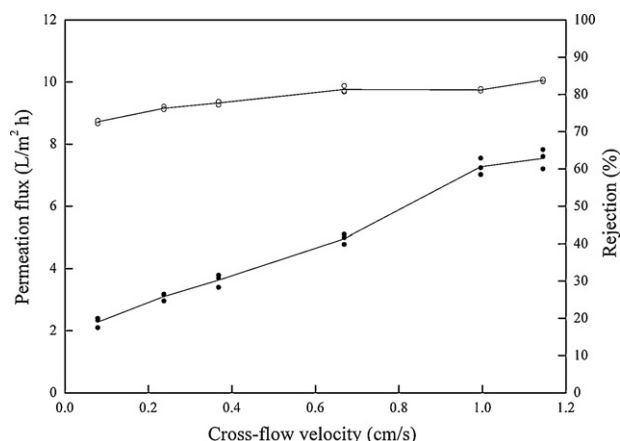


Fig. 11 – Effect of cross-flow velocity of gelatin solutions on permeation flux and rejection (concentration 5 wt%; temperature 55 °C; pressure 0.10 MPa). ●: permeation flux; ○: rejection.

the advantage of increasing the cross-flow velocity was that gelatin did not decompose because gelatin is not a shear sensitive material.

4. Conclusions

When PVP (30K) and acetone were used as mixed additive to prepare PES UF membranes by immersion precipitation phase inversion process, both the pure water flux and rejection (to BSA) were enhanced simultaneously. When the concentration of PES, PVP and acetone in the casting solution was 14, 2, and 1 wt%, respectively, the pure water flux of the prepared membrane reached to 373 L/m² h under 0.1 MPa, and the rejection to BSA was 91%. Compared with other reports, the flux of the PES membrane was high.

When the membrane was used to concentrate gelatin solutions, the rejection values were over 75%. Increasing the feed temperature and the transmembrane pressure enhanced the permeation flux, but the rejection decreased slightly. However, increasing the cross-flow velocity of the feed solution simultaneously increased both the permeation flux and the rejection.

Acknowledgment

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