Influence Of Ethanol Concentration On The Performance Of Polyethersulfone Ultra Filtration Membranes.

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Abstract: In this study, polyethersulfone hollow fiber (PES) ultra filtration (UF) membranes were fabricated using ethanol as additives, N_methyl-2-pyrrolidone (NMP) as a solvent. The water was used as external coagulant. Asymmetric hollow fiber membranes were prepared using wet/dry phase inversion process. Throughout the study, the concentration of PES was fixed at 18 wt % and the ethanol concentration is varied in the range of 0-25wt. %. The performances of membranes were characterized in terms of pore size, pore density measurements, pure water permeation rate (PWP), and solute separation percentage measured by solute polyethylene glycol (PEG).

Keywords: Asymmetric, Polyethersulfone, Ethanol, Solvents.

Introduction

Polyethersulfone is widely used materials for the preparation of microfiltration and ultra filtration membrane (1). PES have favorable characteristics of wide temperature limits, wide Ph tolerances, fairly good chlorine resistance, easy to fabricate membranes in wide variety of configurations and modules, wide range of pore sizes available for UF and MF application ranging from 10A to 0.2µm and good chemical resistance to aliphatic hydrocarbons, alcohols and acids. (2).It has been reported that the properties of polymer membranes could be improved by addition of nonsolvent additives in the dope solution. Kim and Lee investigated the effect of PEG additive as a pore former on the structure formation of membranes and their permeation of thermodynamic and kinetic properties in phase inversion process (3) The several author (4-8) have reported that adding second polymer like polyvinylpyrrolidone, in solutions of polysulfone, polyetherimide and polyvinylchloride produced high porous membranes, good surface properties as well as pore interconnectivity. The addition of organic non solvent in polymer solution could improve the performance and structure of the asymmetric membranes. Wang and co workers reported the precipitation values of several organic non solvents in PSF/Solvent and PES/solvent systems with different temperatures.

Ultra filtration, a novel and powerful pressure-driven separation technology, has been widely used in waste water treatment and food industry (9, 10) to concentrate or fractionate proteins and aqueous solutions. During Ultra filtration, the smaller suspended particle and dissolved molecules pass through the membranes (11, 12), while the bigger molecules are mostly rejected. Some of the rejected molecules adsorbed or deposit on membrane surface causing considerable membrane fouling (13).

In this work, attempt is made to study the effect of ethanol concentration on characteristic and
performance of polyethersulfone ultra filtration hollow fiber membranes using wet/dry phase inversion process. The performances of the membranes are evaluated in terms of pore size, pore density, pure water permeability, and percentage solute (PEG) rejection.

**Experimental**

**Materials used for casting membranes**

Polyethersulfone (PES) was used as polymer for membrane casting. N-methyl-2-pyrrolidone was used as solvent and ethanol used as non solvent additives. Tap water was used as coagulant bath. Polyethylene glycols (PEG) with different molecular weight ranging purchased from Fluka were used as solutes. Feed solutions were prepared using distilled water. Other chemicals used were sodium iodide (KI) purchased from SureChe Products Ltd, barium chloride (BaCl\(_2\)) from Lab guard, iodine (I\(_2\)) from Emory and hydrochloric acid (HCl) from Merck.

**Membrane casting procedure,**

Ethanol and water were mixed separately with N-methyl 2-pyrrolidone in glass bottle. The polyethersulfone dried and added into the bottle, mixed until the solution became homogeneous. The homogeneous low fiber PES membranes were spun at room temperature employing the dry/wet spinning method. The coagulation bath and bore fluid were maintained at room temperature. The fabricated hollow fiber were stored in water bath for 24 hours to remove NMP.After that, fiber were kept in 50wt%glycerol aqueous solution for 24 hour to prevent the collapse of porous structure and finally dried in air at room temperature for different module test. The module were immersed in water for one day to eliminate the effect of glycerol.

**Membrane pore size and pore density**

The pore density and pore size of internal surface of hollow fiber membranes were measured using Optical microscope and calculated as

\[
\text{Pore density}=\frac{N}{A_{\text{in}}}
\]

Where,

- \(N\) is pore numbers of internal surface of hollow fiber membrane while \(A_{\text{in}}\) area of internal surface of hollow fiber membrane (cm\(^2\)). The pore size and pore density of PES hollow fiber membranes having different ethanol concentration are obtained in table 1.

**Measurement of permeate flux and rejection.**

Membrane hollow fiber module was tested for permeate flux and percentage rejection. Initially, experiments were run using distilled water and then, separation experiments were conducted using aqueous solution of 0.01 percentage polyethylene glycol of 10,000 Daltons. All experiment was performed at 1 bar transmembrane pressure and at room temperature. The permeate was collected and its volume was measured to evaluate the membrane performance. System was thoroughly flushed with distilled water between different runs. The concentration of feed and permeate solutions were determined using the analytical method given by Sabde et al. This analytical method involved addition of 1 ml of 5% BaCl\(_2\) in 1 N HCL and 1 ml of solution prepared from a mixture 2% KI (w/v). The color developed was then analyzed using a UV-spectrophotometer at 280 nm against a reagent blank to measure the concentration of permeates. PES Ultra filtration membranes were characterized in terms of pure water permeation (PWP), rejection rates (R), and flux (J). Pure water permeation (PWP) is given by:

\[
PWP= \frac{Q}{PA}
\]

Where \(Q\) is the volume of the pure water permeate (l), \(A\) is the membrane surface area (m\(^2\)), and \(P\) is the transmembrane pressure drop. The solute rejection of the membrane was calculated using the following expression:

\[
R\%=1- \left(\frac{C_p}{C_f}\right) \times 100
\]

Where \(C_p\) is the solute concentration in permeate stream and \(C_f\) is the solute concentration in feed stream.

**Table 1**

<table>
<thead>
<tr>
<th>Ethanol concentration%</th>
<th>Pore size µm</th>
<th>Pore density</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>19.4</td>
<td>6.8×10(^4)</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>16×10(^4)</td>
</tr>
<tr>
<td>15</td>
<td>12</td>
<td>18.9×10(^4)</td>
</tr>
<tr>
<td>20</td>
<td>20.1</td>
<td>12×10(^4)</td>
</tr>
<tr>
<td>27</td>
<td>27.6</td>
<td>3.6×10(^4)</td>
</tr>
</tbody>
</table>

**Table.2** permeate flux and solute rejection for PES hollow fiber membranes.
Table 2.

<table>
<thead>
<tr>
<th>Membranes (H. F) with ethanol conc.</th>
<th>Permeate flux (L/m² h Bar)</th>
<th>Solute Rejection % (PEG10,000)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>47</td>
<td>83</td>
</tr>
<tr>
<td>10</td>
<td>99</td>
<td>92</td>
</tr>
<tr>
<td>15</td>
<td>103</td>
<td>84.7</td>
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<tr>
<td>20</td>
<td>113</td>
<td>22.9</td>
</tr>
<tr>
<td>27</td>
<td>167</td>
<td>14</td>
</tr>
</tbody>
</table>

Result and discussion

In order to study the effect of ethanol concentration on hollow fiber membranes, Fig. 1 shows that pore size first decreased with increased ethanol concentration from 0 to 15 wt% and then increased with increased in ethanol concentration from 15 to 27 wt%, whereas for Fig. 2, the pore density first increased with increased in ethanol concentration from 0 to 15 wt% and then decreased with increased in ethanol concentration from 15 to 27 wt%. The PES hollow fiber membranes made with NMP and water as internal coagulant has very closed to binodal on the solvent /nonsolvent axis of ternary phase diagram for PES and hence gelation is not favored phase separation mechanism, the microvoids are open ended on inside. The delay coagulation in surface region of hollow fiber membranes and the ethanol present low concentration resulted into fine pore formation. Therefore internal surface affected strongly with ethanol concentration. The pore size could be adjusted with controlling the ethanol concentration in dope solution.

Effect of ethanol concentration on permeate flux and rejection.

The Figure 3 shows that permeate flux increased with increased in ethanol concentration. The permeate flux increased from 47 to 164 (L/h.m².bar) with increased in ethanol concentration from 0 to 27%. The Fig. 4 shows that rejection percentage increased with increased in ethanol concentration from 0 to 10 wt%, further increased in ethanol concentration beyond 10 wt%, rejection percentage decreased. With an increase of ethanol concentration from 0 – 10 wt%, internal surface becomes more microporous while the pore size reduces. However, the microporous structure in the internal surface become less while the pore size increases with an increase of ethanol concentration from 10 to 27 wt%.

Fig. 1.

Fig. 2.

Fig. 3.

Fig. 4.
**Conclusion.**

Polyethersulfone ultra filtration membranes were spun by wet/dry phase inversion process. The polymer solution were prepared using 18wt% PES in NMP and different concentration of ethanol. PES hollow fiber membranes could be controlled with addition of different concentration of ethanol. The experimental result shows that pure water permeability increased with increased in ethanol concentration whereas rejection% highest at 10wt% ethanol concentration for solute PEG of 10,000Dalton. In this topic, we might be prepared polyethersulfone ultra filtration membranes with high pure water permeability and molecular weight cut off of the membranes approximately 10,000.

**References.**


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